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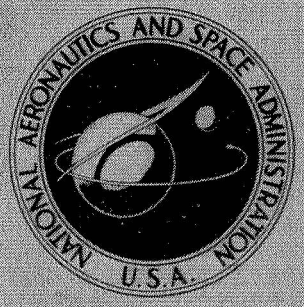

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CONTAMINATION OF REFRACTORY METALS BY OXYGEN, WATER VAPOR, AND CARBON MONOXIDE

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Prepared by
GENERAL ELECTRIC COMPANY
Schenectady, N. Y.
for Lewis Research Center

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16. Abstract

The sticking probabilities were determined for oxygen, water vapor, and carbon monoxide on columbium 1% zirconium, pure tantalum and several combinations of tantalum alloyed with tungsten and hafnium. The investigation was made with the metals exposed to temperatures between 1158 and 1373° K and with gas pressure between approximately 10^{-5} and 10^{-7} torr. The specimen weight increase due to absorbed gas was calculated from the specimen gettering rate. The results closely agreed with the weight gains determined by gravemetric analysis and gas analysis.

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FOREWORD

The work described herein was performed under National Aeronautics and Space Administration Contract NAS3-8518 and was originally issued as General Electric Report S-69-1114. Initial conceptual design and experimental work were done at the General Electric Company Research and Development Center, Schenectady, New York. The test specimens were evaluated and the data analyzed at the NASA-Lewis Research Center, Cleveland, Ohio. Mr. R. A. Lindberg, Space Power Systems Division, NASA-Lewis Research Center, was the project Manager.

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1.0 SUMMARY

The purpose of the work carried out under this contract was to expose columbium and tantalum alloy samples to high temperatures in an atmosphere of low-pressure contaminant gas in order to determine some of the gas absorption processes of the metal. The metal samples were columbium 1% zirconium, pure tantalum and tantalum specimens alloyed with various combinations of tungsten and hafnium. The metals were exposed to temperature between 1158 and 1373^{o}K and to contaminant gases with pressures between approximately 10^{-5} and 10^{-7} torr. The contaminant gases were oxygen, water vapor and carbon monoxide.

The sticking probability for several combinations of contaminant gas and alloy composition were determined under various temperature and pressure conditions. The sticking probability was found to be independent of specimen temperature over the experimental temperature range.

The specimen weight increase due to absorbed gas was calculated from the specimen gettering rate. The results closely agreed with the weight increases determined by a gravimetric analysis and gas analysis.

2.0 INTRODUCTION

The program described by this report was conducted to determine the oxidation rate or gettering rate for oxygen, water vapor and carbon monoxide of columbium, and tantalum alloy specimens under conditions of low pressures and high temperatures.

The gettering rates were established by the method of measuring the pressure drop across a known flow conductance, from which the gas mass flow rate (equal to the gettering rate) can be computed. A knowledge of the pressure directly over the specimen, plus knowledge of the gettering rate can then be used to calculate the "sticking factor" (that fraction of total molecules striking the specimen, which do not rebound). The sticking factor is the item of primary interest in these experiments.

A total of 12 exposures were conducted during the program, with oxygen, water vapor and carbon monoxide pressures at levels ranging from about 10^{-5} torr to 3×10^{-7} torr and temperatures at levels of 885^{o}, 980^{o} and 1100^{o}C.

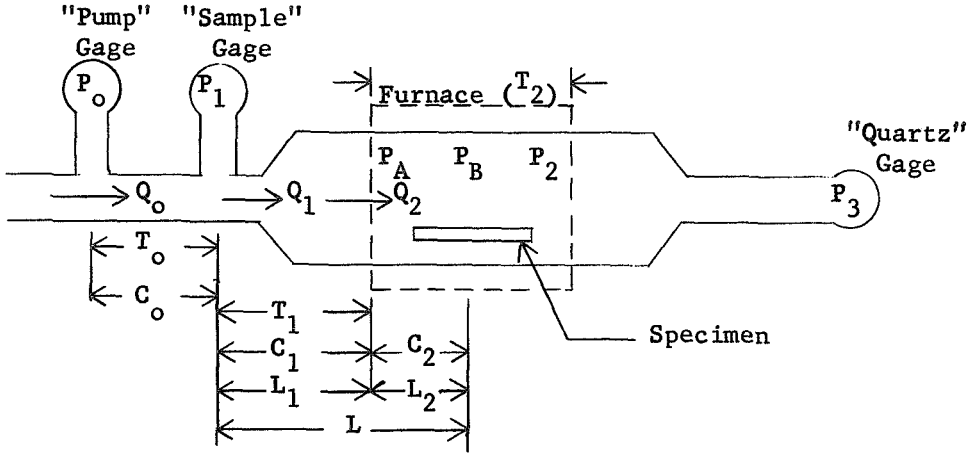
The 10^{-10} torr vacuum system used on contract NAS 3-4169 was modified to increase the reliability, accuracy and simplicity of operation. A new system was designed and constructed to incorporate a new pumping system, facilitate introduction of not only oxygen but water vapor and carbon monoxide plus the ability to measure partial pressure at the same three points that the total pressure was being measured by ion gages. In addition to the 12 exposures, the program included several calibrations of both the ionization gages and partial pressure analyzers.

The scope of the investigation was confined to exposing the specimens to the various environments and documenting the data and experimental conditions. Subsequent evaluation of the exposed specimens by chemical and metallurgical techniques and analysis of data were conducted at the Lewis Research Center of

the National Aeronautics and Space Administration. The work performed by the General Electric Company was done at their Research and Development Center in Schenectady.

3.0 NOMENCLATURE

The sketch below and the following list will define the symbols used.



A = Conductance tube cross-sectional area (cm^2)

a = Specimen surface area

B = A constant; see Section 11.3

C = Conductance, defined by $Q = C \Delta p$ (liters/sec)

D = Internal conductance tube diameter (cm)

E = Sticking Probability

G = Mass flux ($\mu g\ sec^{-1}\ cm^{-2}$)

K = Clausing factor, a function of L/D = .0694

L = Length, along tube axis (cm)

$\dot{m}$ = Mass flow rate (μg/sec)

M = Molecular weight of gas (g/mole)

P = Pressure (torr)

Q = Flow rate in terms of pressure x volume flow rate (proportional to mass flow rate at a given temperature) (torr liters/sec)

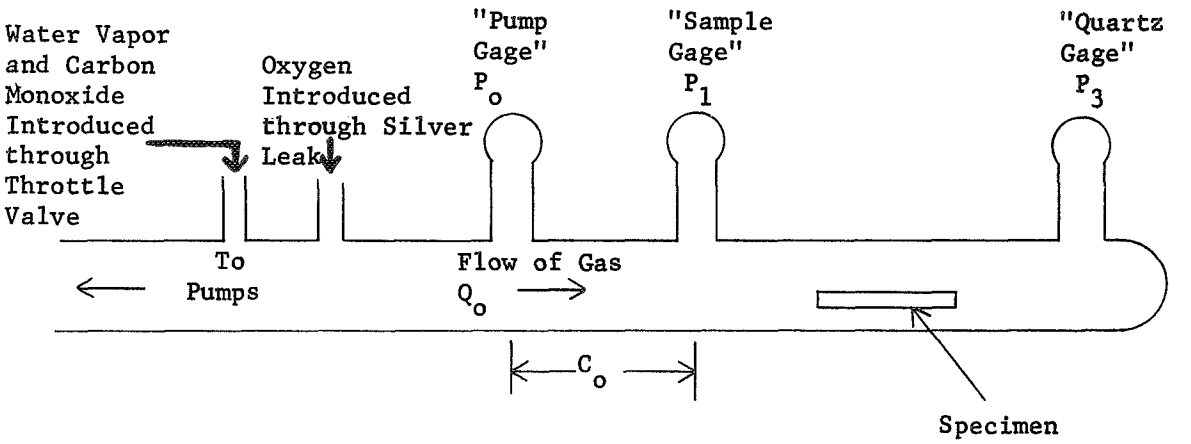
R = Gas constant (62.3 torr liter $mole^{-1}$ deg K^{-1}) or (8.31×10^7 dyne cm $mole^{-1}$ keg K^{-1})

T = Absolute temperature (deg K)

V = Volume flow rate (liters/sec)

4.0 EXPERIMENTAL APPARATUS

The schematic below indicates the principle used to determine gettering rates.



The gas flow rate, Q_o, is determined by measuring the pressures, P_o and P_1 and then using the equation

$$Q_o = C_o (P_o - P_1)$$

where C_o is the known flow conductance between pressure gages. In the systems, this conductance consists of a right circular cylinder of accurately measured dimensions, from which C_o is calculated.

Details on the construction of the flow conductances and calculations of C_o are given in the Appendix, Section 11.3.

The ion gage measuring pressure P_3 is used, with a thermal transpiration correction,* to determine the pressure directly over the specimen, P_2. This pressure is needed to establish the sticking factor.

---

* For example of thermal transpiration correction, see Appendix, Section 11.4.

4.1 Description of System

The system is shown schematically in Figure 4.1 with dimensions of various elements given. Not shown is the pumping system, which consists of a 40 liter/sec ion pump, a sorption pump and a forepump.

Physically, the system is mounted on a table 9' x 33" x 36" and insulated by 2" thick J-M Marinite - 23. All instrumentation and pumping apparatus are mounted under the table top for effective positioning and compactness. Photographs of the Refractory Metal Contamination System are given in Figures 4.2 and 4.3. Figures 4.4 and 4.5 show large bake-out furnace positioned on top of table in readiness of bake-out of entire system. Strip heaters are used to produce a 350°C bake-out temperature.

Introduction of oxygen to the system was accomplished with a heated silver permeation membrane. It was found that with constant specimen gettering rate and with approximately one atmosphere of 0_2 applied to the membrane, the system pressures held constant for many days. The silver membrane leak was found to be relatively insensitive to small 0_2 pressure changes on the high pressure side.

Introduction of the water vapor and carbon monoxide was accomplished by containing the contaminating gas in a small vessel. The gas was then admitted into the vacuum system through a throttle valve.

The vacuum system usually reached the low 10^{-8} torr range after bake-out, before the contaminating gas was admitted.

The specimen was brought up to temperature by a resistance heated furnace. The furnace also heated the quartz walls surrounding the specimen. This is designated a Hot Wall Test where wall temperature and specimen temperature are the same.

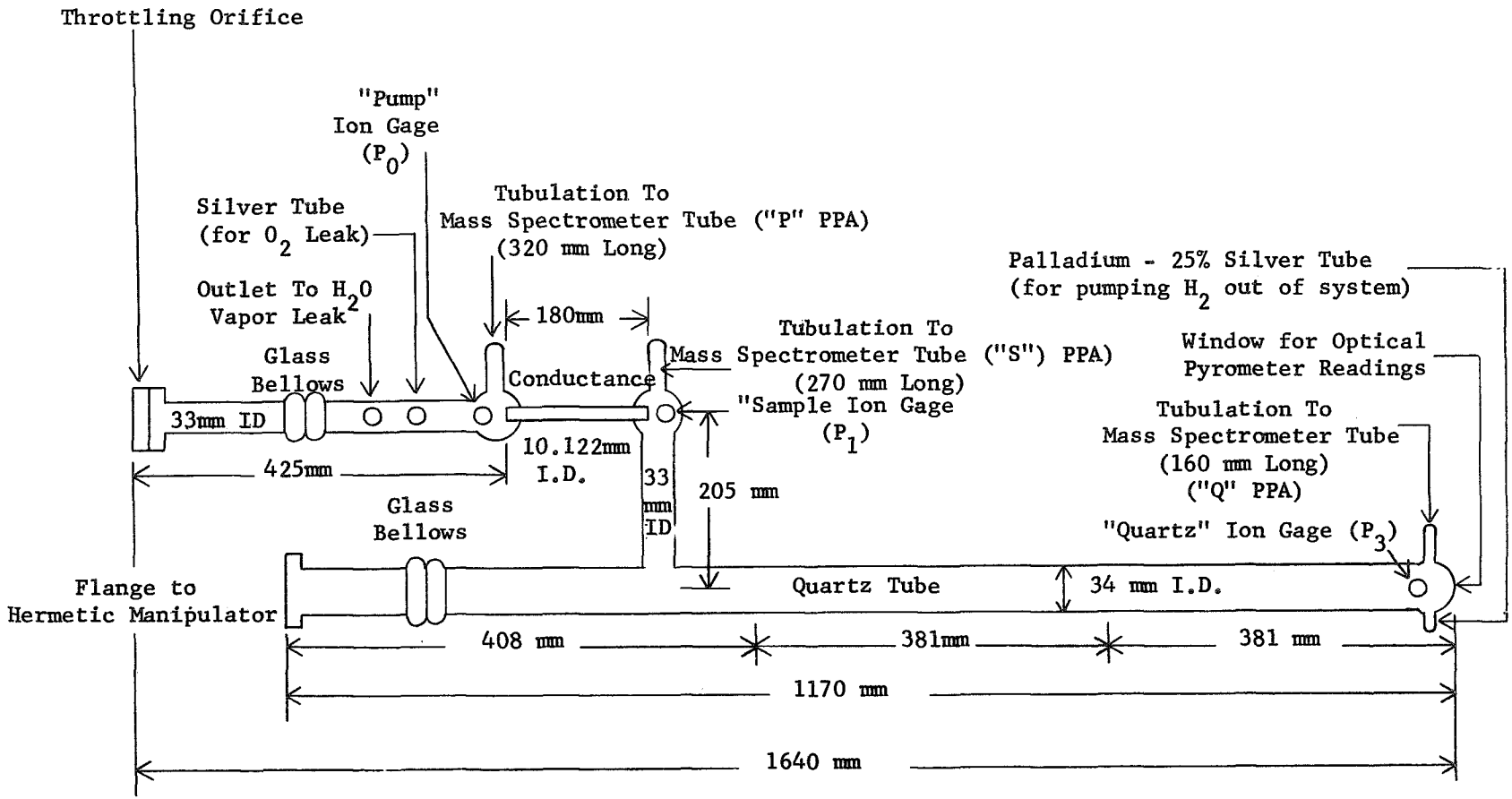


Figure 4.1 Refractory Metal Contamination System Schematic

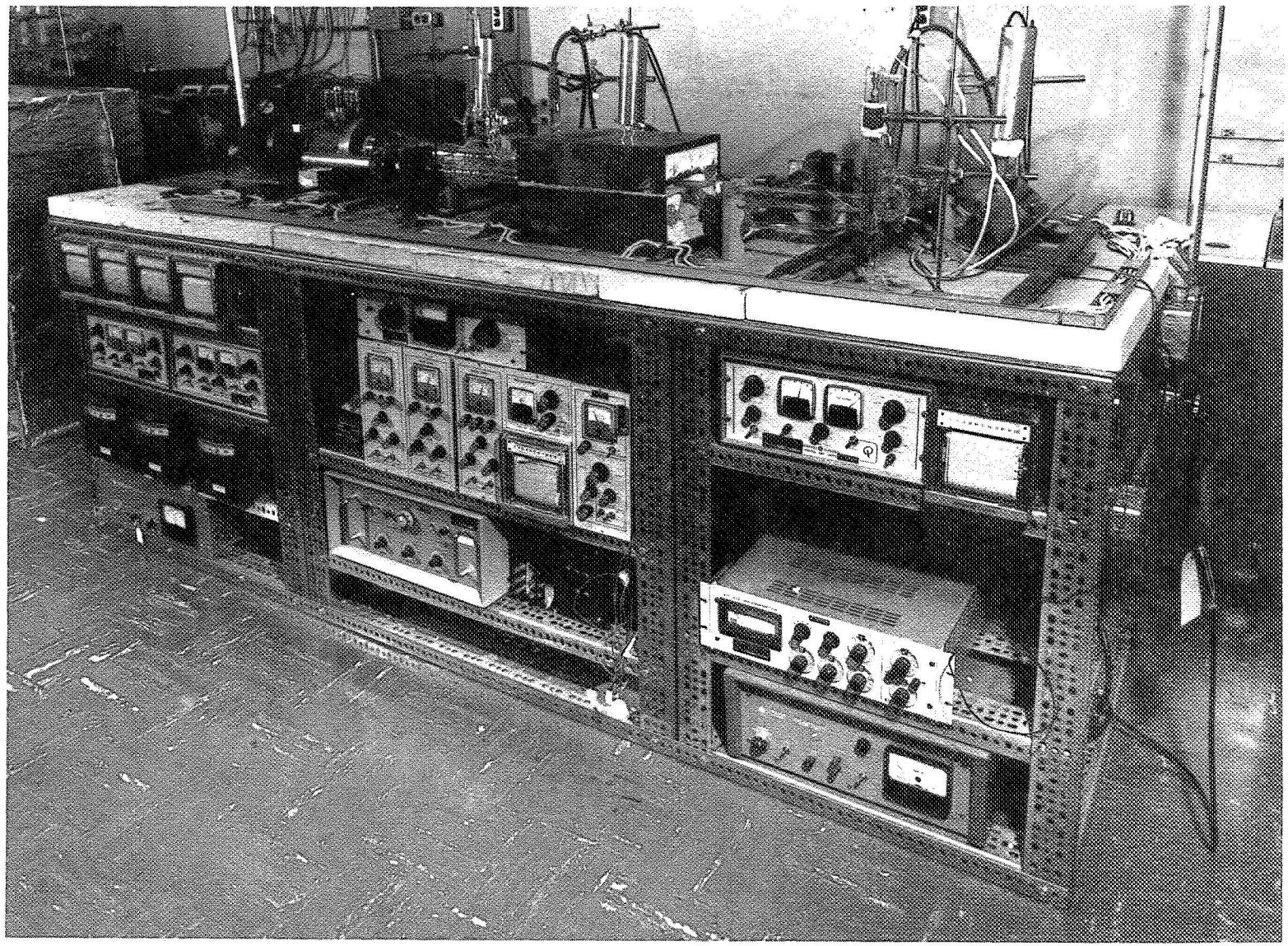


Figure 4.2. Refractory Metal Contaminations System

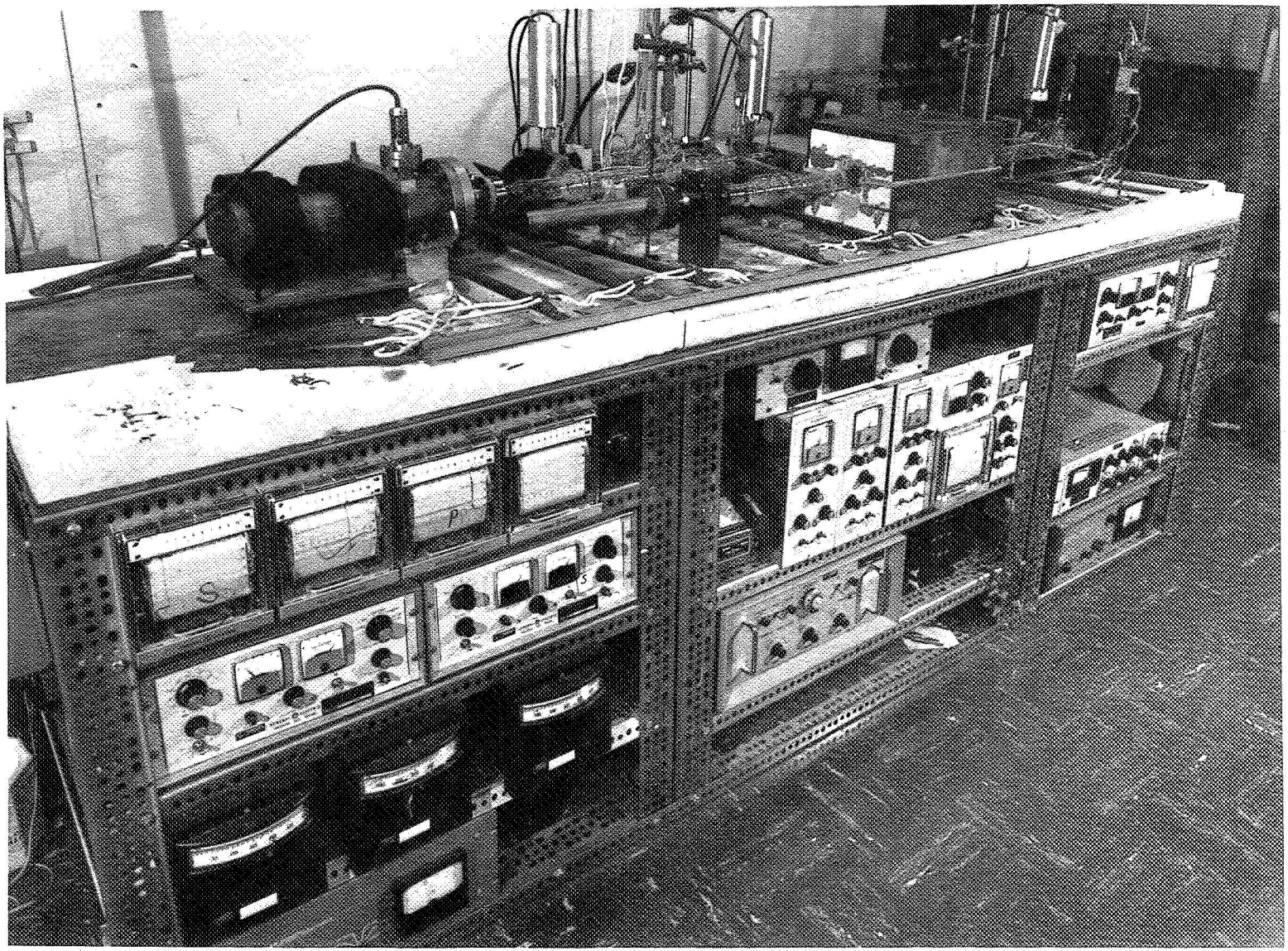


Figure 4.3. Refractory Metal Contaminations System

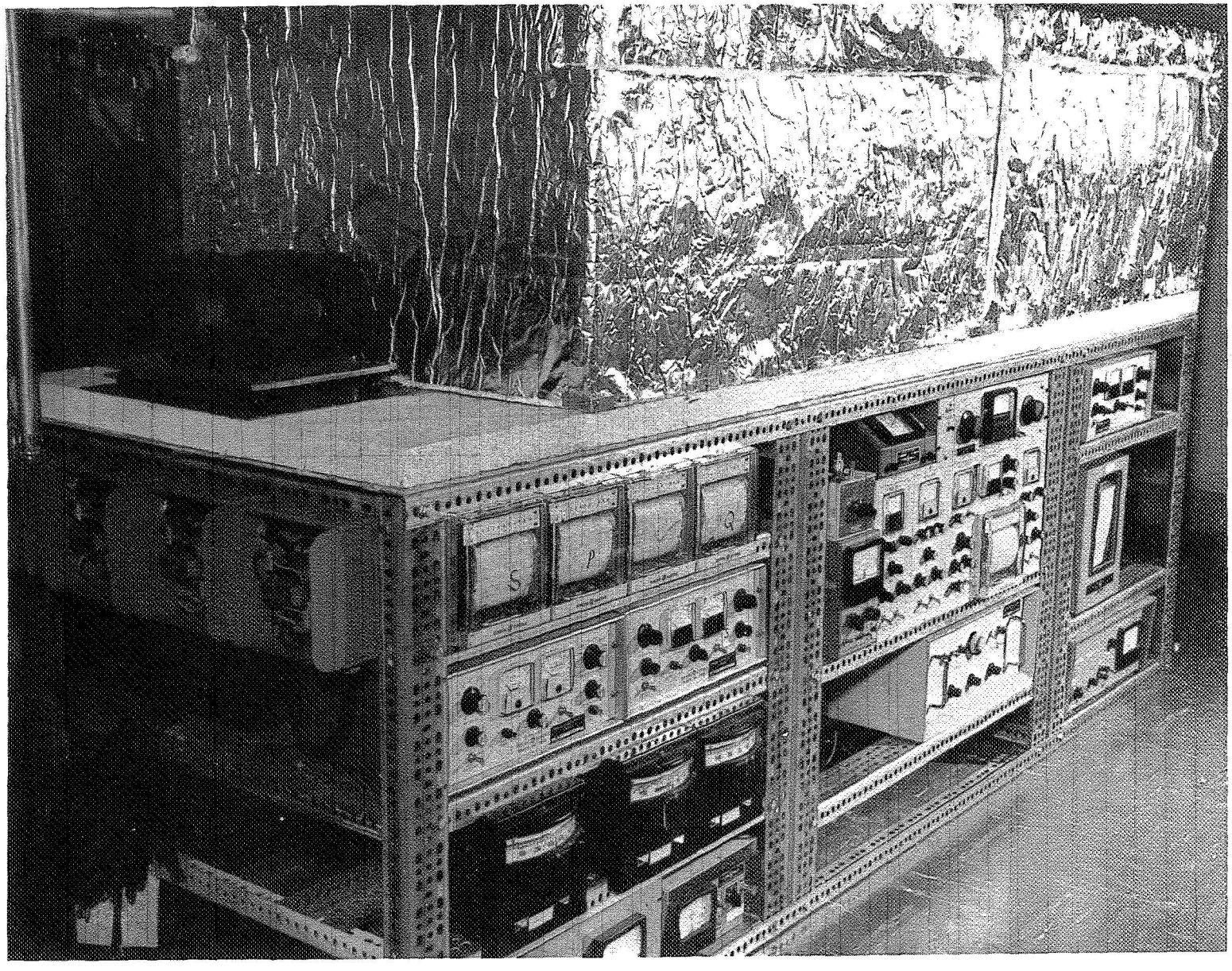


Figure 4.4. Bake-out Furnace Configuration

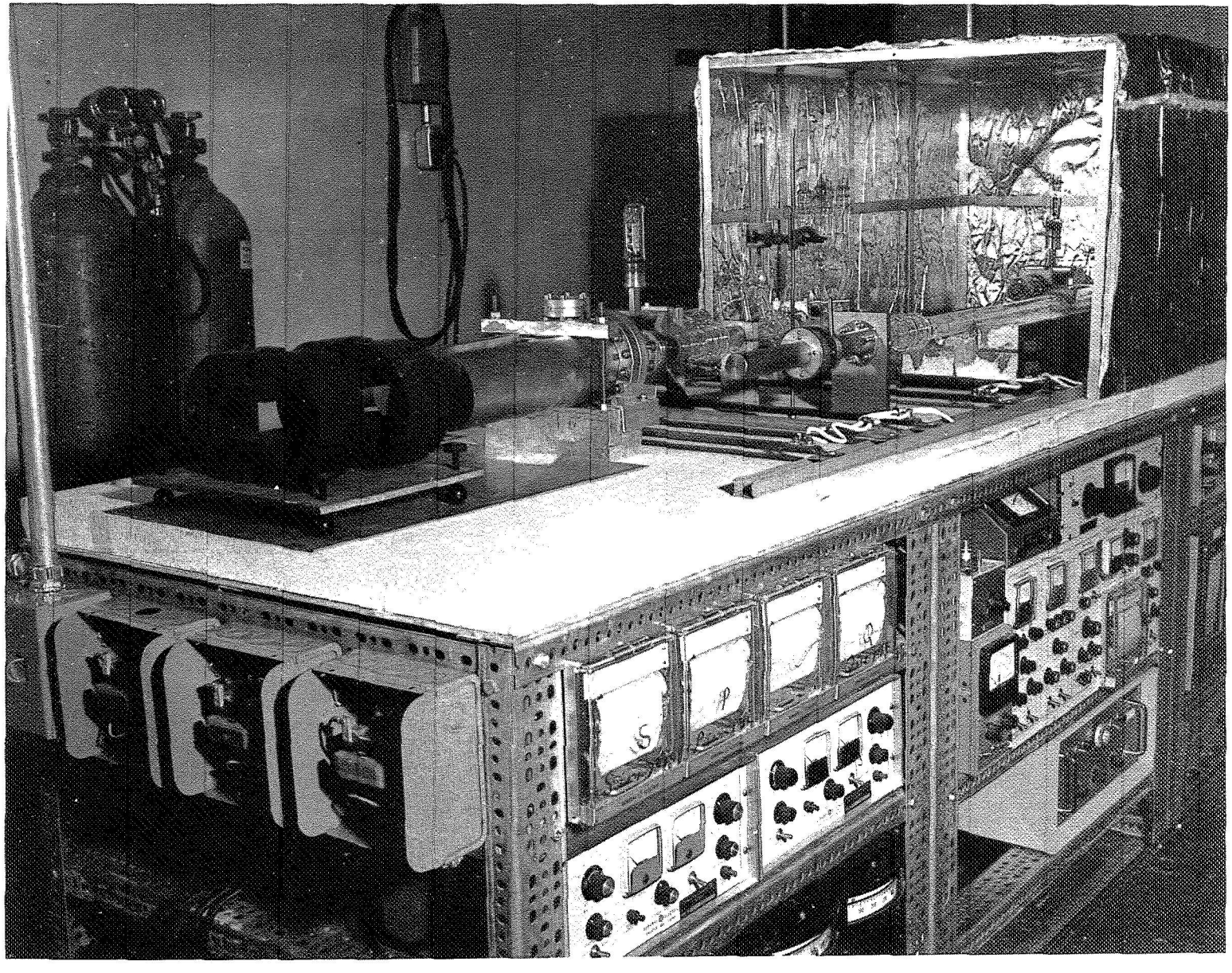


Figure 4.5. Section of Bake-out Furnace

4.2 Instrumentation

Ion Gages

The ion gage tubes* contained one thoria-coated iridium filament and one tungsten filament. Only the thoria-coated filaments were used. These filaments were preferred to the tungsten because of longer life and a reduction in the rate of conversion of O_2 to CO at the filament. The ion gage tubes were operated at an emission current of 1 mA. The ion gage readings were recorded on strip chart recorders.

"Secondary Standard" Ion Gages

Two "secondary standard" ion gages were used to calibrate the three ion gages mentioned above. The calibration of these standard ion gages is given in Appendix, Section 11.5. An independent laboratory** performed the calibration of these gages.

Mass Spectrometers

The three mass spectrometer tubes*** used to determine gas composition were glass analyzer tubes with thoria-coated iridium filaments.

One sweep module supplied the accelerating voltage from a common circuit with built-in timer to the three partial pressure analyzers. Three independent emission controls and drawout voltage circuits completed the electronic control of the mass spectrometers.

Three picoammeters were used in conjunction with the partial pressure analyzers. Additional readout equipment consisted of three strip chart recorders. The partial pressure analyzer tubes used three-kilogauss bakeable permanent magnets.

---

*General Electric Co. Model 22GT-102
**AVCO Corp., Wilmington, Mass.
***General Electric Co. Model 22RT-103

Ion Pump Controls

A high voltage power supply* was used for the 40-liter/sec ion pump.

A pump control unit** was used for the 5-liter/sec ion pump that pumped hydrogen away from the controlled hydrogen leak during injection of water vapor into the vacuum system.

Controlled Leaks

A permeation oxygen leak was used to introduce oxygen to the system. This controlled leak was powered by a d.c. power supply to provide a stable oxygen leakage. A permeation hydrogen leak was used to remove hydrogen from the system during the reaction of the test specimens with water vapor.

The introduction of water vapor and carbon monoxide was made through a throttle valve.

Temperature Measurement

Measurements of specimen temperature were made by a optical pyrometer.***

The pyrometer was calibrated prior to the start of the program. It was calibrated against a standard tungsten filament which has been calibrated by the N.B.S., and which had an uncertainty of ± 3 deg. C in the 900 to 1100 deg. C range. The pyrometer reading was found to be within ± 3 deg. K of the "brightness temperature" of the standard filament throughout the range of interest.

The Thermophysical Properties Research Center at Purdue University supplied a value (under TPRC Serial No. 28741) for spectral emissivity of columbium at .65 microns of .42 (a value which holds quite well over the range 1100 deg. K to 1500 deg. K). The value of .42 when corrected for glass losses gave an effective emissivity of .39. Selected values from the correction table are presented in Table 4.1

* Varian Associates, Model V-7410-B
**Varian Associates, Model V-1304
***Leeds and Northrup Cat. No. 8622-C

Table 4.1 Pyrometer Temperature Corrections

Observed Temperature		True Temperature		
Deg. K	Deg. C	Deg. K	Deg. C	Correction
1157	884	1216.98	943.98	59.98
1252	979	1322.54	1049.54	70.54
1372	1099	1457.17	1184.17	85.17

Specimen Manipulator

A hermetic push-pull-rotary manipulator* was used to position specimens in and out of the quartz tube furnace. Its seal to the system is made by a standard 2-3/4 inch O.D. flange thus enabling easy replacement of samples.

Bake-out Furnace

A bake-out furnace consisting of two equal parts having dimensions of 47" x 33" x 24" was constructed of 1/16" aluminum and attachment to this frame a Carborundum Fiberfrax Lo-Con blanket 1/4" thick with aluminum foil backing on both sides.

Conductance Tube

A precision bore tubing** having dimensions of 180 mm length, 10.122 mm I.D., and heavy wall was used on the system. This is surrounded by 2 1/2" pyrex tubing with glass bellows to prevent breakage during bake-out. A photograph of the conductance tube is shown in Figure 4.6.

Throttling Orifice

A 2 3/4" O.D. blank-fixed type flange*** was modified by putting a knife edge on the blank side and drilling a 0.152" hole through the center over a length of 0.276". This modification enabled the pumping speed to be cut down

*Physics Technology Laboratory
**Wilmod Glass Co.
***General Electric Co. Model 22HF-112

from 40 liters/sec. to approximately 1 liter sec.

Specimen Furnace

A heavy duty tube furnace was used to obtain specimen temperatures in the ranges of 885°C, 950°C, 980°C, and 1100°C.

Quartz Sled

A quartz sled was made to hold the specimen in approximately the center of the furnace. A photograph of the sled is shown in Figure 4.7.

Bakeable Valve

A bakeable valve (6 liter/sec conductance) was used to close off rough pumping system from high vacuum system.

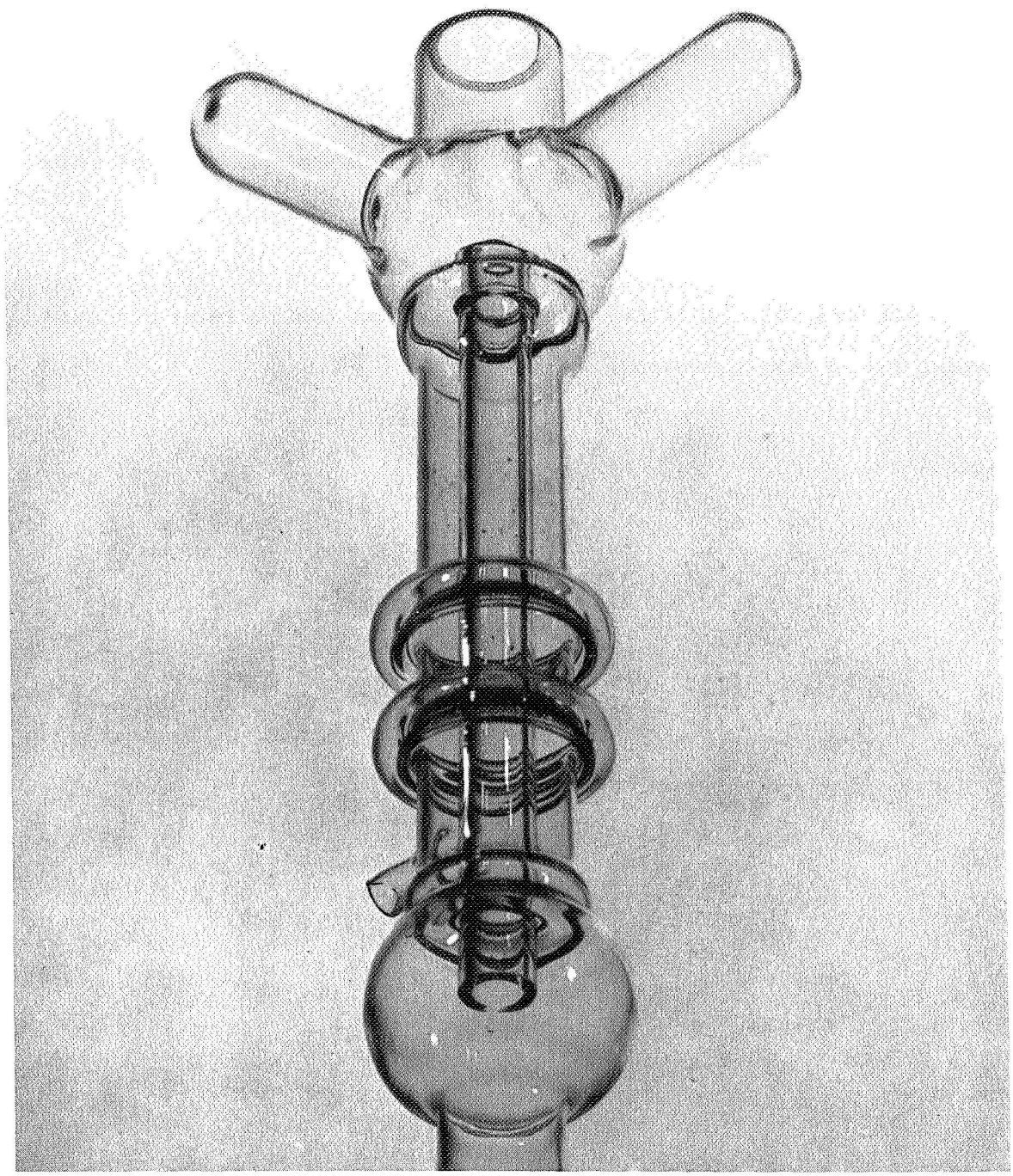


Figure 4. 6. New Design Conductance Tube

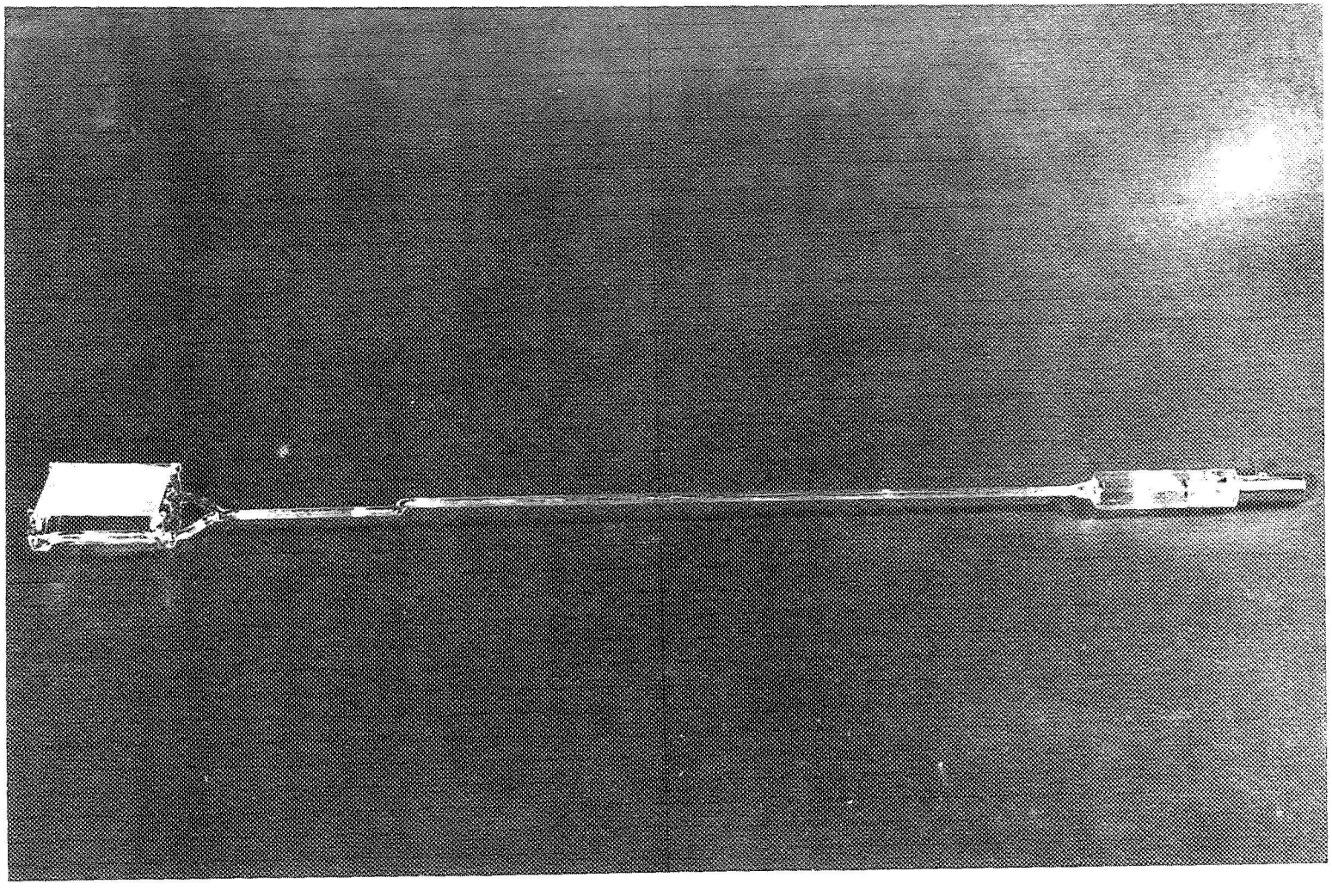


Figure 4.7. Specimen Holder

5.0 EXPERIMENTAL PROCEDURES

A condensed list of steps in preparing the apparatus and conducting the exposure is given below.

5.1 Preparation of Apparatus

(a) The first step in preparation was to load the specimen on its sled and insert the sled onto the hermetic manipulator which is shown in Figure 5.1. Initially, the specimen was located on the sled near the tubulation opening to the P_1 ion gage so that the specimen furnace could subsequently be raised in temperature (see step (f) below) while the specimen still remained cool. The specimen was located with identifying numbers facing upwards and at the end toward the P_3 ion gage.

(b) The second step was to evacuate the entire system with a forepump in series with a sorption pump to a pressure of approximately 1-2 microns.

(c) The system was then checked thoroughly for leaks with a helium mass spectrometer leak detector*.

(d) The bake-out furnace was placed over the apparatus as shown in Figures 4.4 and 4.5 and the system was baked-out at between 300 and 350^{o} for 16 to 24 hours.

(e) The furnace was removed after cooling to below 100^{o}C and the bakeable valve closed. This separated the system from the pumping apparatus mentioned in step (b). At this point, the 40 liter/sec ion pump was turned on (Figure 5.2) and the system was pumped down to 10^{-6} range.

(f) The ion gages were connected and set at an emission current of 1 ma. The specimen furnace was turned on and brought up to a temperature of 1100^{o}C.

*General Electric Co., Model LC-20

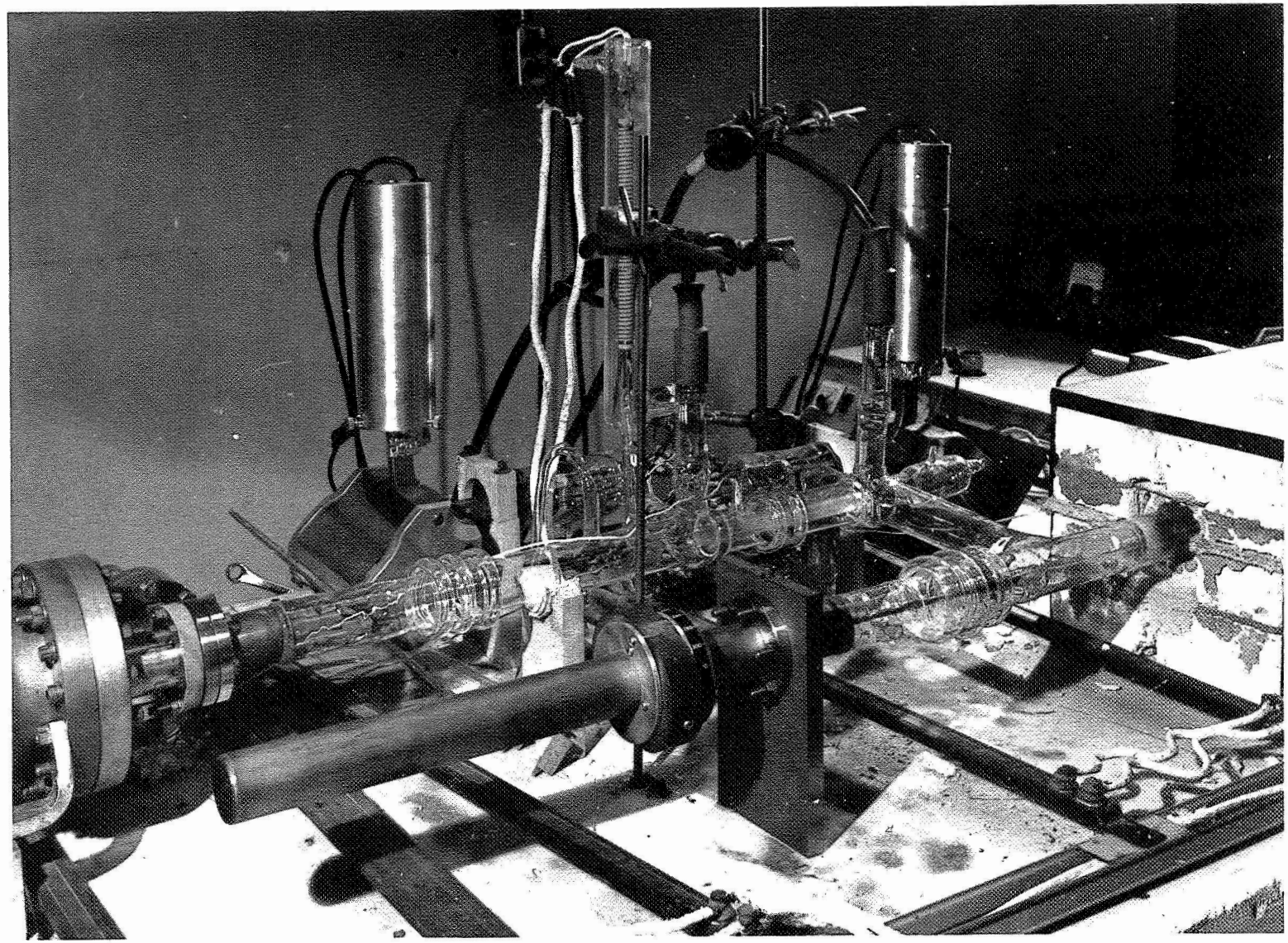


Figure 5.1. Hermetic Manipulator

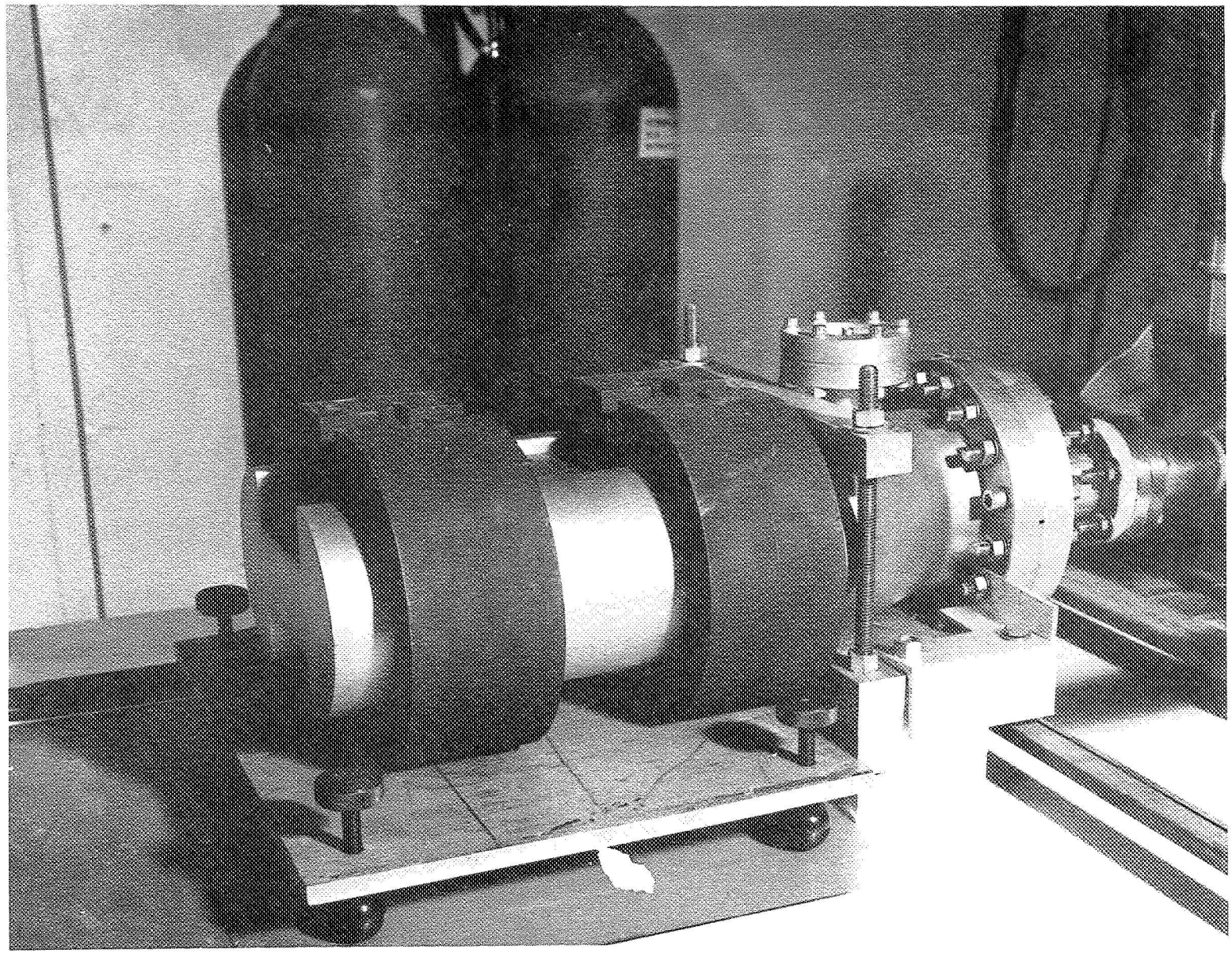


Figure 5.2. Ion Pump

(g) The ion gages were then degassed. An emission current of approximately 30 ma for 15 minutes was usually used in the degassing procedure.

(h) The mass spectrometers were turned on. The emission current was set at 1 ma and the voltage at 70 volts.

(i) The system was then run until the ion gages reached the low 10^{-8} torr range.

(j) The specimen was then pushed into its final position at the center of the furnace by the magnetically controlled hermetic manipulator. Pushing the specimen into the furnace usually produced a burst of gas, raising the pressure suddenly. This increase in pressure was identified as hydrogen coming from the specimen. This occurs even through all the specimens were vacuum annealed at $3600^{o}F$ for 1/2 hour and at 10^{-7} torr prior to test.

(k) Pumping generally continued until the P_o pressure went back to nearly the same level as before step (j). This usually required 12 to 15 hours. At this time, the system was ready for introduction of the contaminating gas.

5.2 Conducting Exposure

Oxygen pressure was regulated by controlling the temperature of the silver leak. Prior to admitting oxygen, the silver leak was outgassed by pumping a vacuum on the upstream side and heating. Oxygen was then admitted to the upstream side of the leak (at time = 0 for the exposure) and maintained at approximately 5 inches of water above atmospheric pressure for the remainder of the exposure. After admitting oxygen it generally took about 15 minutes to

reach the correct pressure decade and another 15 to 30 minutes to level off at the final pressure. No measurement was taken of the silver leak temperature.

Water vapor and carbon monoxide pressure were regulated by the use of a throttle valve. Prior to admitting the water vapor, the glass vessel containing the water vapor was outgassed by pumping a vacuum on the upstream side and heating. After admitting the distilled water to the vessel, the water was first frozen and then boiled under vacuum to remove any trapped gas. The vessel was then sealed off and the water allowed to stabilize to an equilibrium pressure. Before admitting the carbon monoxide, the glass vessel was again outgassed as described above and backfilled with carbon monoxide to an equilibrium pressure of approximately 5 psi. Equilibrium pressures were achieved in similar times as those described for oxygen upon admission into the system.

All ion gage pressures were recorded continuously on strip chart recorders. Mass spectrometer readings were also recorded continuously on strip chart recorders. This was done by setting a sweep time of 30 minutes which allowed a spectrum of the gases of interest, namely CO_2, O_2, CO, He and H_2 to be recorded every half hour at three locations during the entire run. At the end of an exposure, the glass system was allowed to cool down to ambient and then the pressure was brought up to atmospheric. The glass system was then opened up, the specimen removed and the specimen sent to the NASA Lewis Research Center for analysis. The strip chart recorder rolls were also sent to NASA for use in reducing the data.

5.3 Test Specimens

The characteristics of the various test specimens used during the course of the experiments are given in Table 5.1. The first section of Table 5.1 lists

Table 5.1 Test Specimen Characteristics

Specimen	Thickness (cm)	Interstitial O_2	Interstitial N_2	Analysis*(ppm) C	Analysis*(ppm) H	Surface Finish Microinches	Sample	Run
Columbium								
-.75% Zr	.127	978	11	17	1	60	1	3
-.89% Zr	.113	146	20	10	1	42	7,5	1,2
-.89% Zr	.113	172	19	5.5	1	42	1,2	4,5
Tantalum	.079	22	17	3.5	1	31	77,76,78	6,11,12
Ta -1.81% Hf	.076	16.5	13.3	9.5	1	25	1	9
Ta -10.19%W	.081	49	11	2	1	19	80	8
Ta -8.01%W -1.88%Hf (T-111)	.076	22	9	2.5	1	10	84,85	7,10

Run No.	Gas	Sample	Dimensions (cm)	Surface Area (cm^2)	Final Weight (gms)	Final Analysis (ppm)	No O_2 Analyses	Con /sec
1	O_2	7	3.861 x 2.629	21.7499	9.58683	431.5-O_2	6	.6315
2	O_2	5	3.861 x 2.591	21.4636	9.63449	5,993-O_2	6	.6315
3	O_2	1	1.290 x 2.527	7.49173	3.51002	12,150-O_2	2	.6315
4	H_2O	1	3.851 x 2.591	21.4086	9.59003	1,770-O_2	5	.8420
5	H_2O	2	3.830 x 3.644	21.7194	9.67419	2,173-O_2	6	.8420
6	H_2O	77	3.818 x 2.672	21.4240	13.54613	1,088-O_2	6	.8420
7	H_2O	84	3.962 x 2.654	21.7499	13.60089	1,618-O_2	6	.8420
8	H_2O	80	3.957 x 2.619	21.7921	13.79731	1,046-O_2	6	.8420
9	H_2O	1	3.843 x 2.507	20.2365	12.72418	1,408-O_2	6	.8420
10	H_2O	85	3.820 x 2.685	21.5542	13.63618	3,125-O_2	6	.8420
11	CO	76	3.818 x 2.670	21.4042	13.55971	179-O_2,120-C	6	.6751
12	CO	78	3.818 x 2.667	21.3844	13.50483	183-O_2,122-C	6	.6751

* After vacuum annealing 30 minutes at approximately $3600^{o}F$ and 10^{-7} torr.

the important chemical characteristics and the second section lists the important physical properties.

The 30 minute annealing process at $3600^{o}F$ and 10^{-7} torr was an attempt to remove as many interstitial contaminants as possible before the sample was subjected to the contamination exposure. The combined conditions of temperature and pressure were more extreme during the annealing process than during the contamination exposure. Therefore, volatile interstitial components should be expelled during annealing rather than during the exposure.

6.0 EXPERIMENTAL CONDITIONS OF EXPOSURES

6.1 General

Twelve exposures were conducted during the program. These were of two types: (1) A "steady state" exposure in which the pressure and temperature of the specimen were held constant for a 236 hour period (approximately 10 days), and (2) A "step" exposure in which the pressure and temperature were held constant for approximately 24 hours, after which they were changed to new values for the next step or phase of the exposure. The "step" exposures generally consisted of temperature steps of 1100, 980 and 885°C at pressure levels of 10^{-5}, 10^{-6}, 10^{-7} torr, resulting in nine steps to complete the run. A list of the twelve runs is presented in Table 6.1. It includes type of run, contaminating gas, ion gage set and partial pressure analyzer set. Two steady state runs were also used to derive the ion gage sensitivity for the contaminating gas.

The step runs started at the high temperature (1100°C) and the high pressure (10^{-5}) and proceeded in the following manner:

Pressure	Temperature
10^{-5}	1100°C
10^{-5}	980°C
10^{-5}	885°C
10^{-6}	885°C
10^{-6}	980°C
10^{-6}	1100°C
10^{-7}	1100°C
10^{-7}	980°C
10^{-7}	885°C

Table 6.1

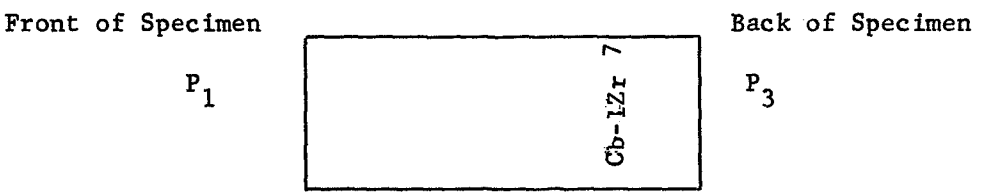
Run	Specimen	Type of Run	Contaminating Gas	Ion Gage Set	PPA Set
1	Cb-Zr #7	Steady State	Oxygen	#2	#1
2	Cb-1Z #5	Step	Oxygen	#2	#1
3	Cb-1Z #1 (Short Sample)	Step	Oxygen	#3	#2
4	Cb-1Zr #1	Steady State	Water Vapor	#3	#2
5	Cb-1Zr #2	Step	Water Vapor	#3	#2
6	Ta - #77	Step	Water Vapor	#3	#2
7	T-111 #84	Step	Water Vapor	#3	#2
8	Ta-10W #80	Step	Water Vapor	#3	#2
9	Ta-2Hf #1	Step	Water Vapor	#3	#2
10	T-111 #85	Steady State	Water Vapor*	#3	#2
11	Ta #76	Steady State	Carbon Monoxide*	#3	#2
12	Ta #78	Step	Carbon Monoxide	#3	#2

*Used in Ion Gage Sensitivity Derivations

6.2 Index of Exposure Data and Discussion

Run # 1

Run # 1 was a "steady state" run using oxygen as the contaminating gas and sample Cb-1Zr #7. The position of specimen in the system and data concerning the run are discussed below.



The face shown in the Sketch is the top of the sample as it was in the quartz tube. The inscription Cb1Zr 7 is on the edge closest to the P_3 ion gage. The distance from the P_1 ion gage to the front of the specimen was 11 1/4". The distance from the P_3 ion gage to the back of the specimen was 22 1/8". Refer to Figure 4.1.

The position of the specimen in the quartz tube and the distances from the edges to P_3 ion gage and P_1 ion gage remained the same for all runs except for run #3 which will be depicted separately. The average temperature during the run was 977.77°C. The pressure was held at 3×10^{-7} on the P_1 ion gage. Total time in which the specimen was exposed to O_2 was 242 hours and 45 minutes. The second set of ion gages were used for this run and their calibration curves are in Section 11.1, Calibration of Ion Gages. The first set of partial pressure analyzer calibrations and sensitivity values were used for this run and can be found in Section 11.2 Partial Pressure Analyzer Calibrations.

The three partial pressure analyzers are identified by their location, namely "P" for P_o location, "S" for P_1 location and "Q" for the P_3 location.

During the run a double oxygen peak effect was noted on the "P" partial pressure analyzer and several tests were conducted after the run. As a result of tests discussed below, it was concluded that the large peak during the run was oxygen and the smaller peak was an unknown derivative of oxygen having a slightly higher mass number. It appeared that this derivative is oxygen dependent rather than ion pump dependent. The following is a brief description of the tests conducted. Before admitting oxygen to the system, there were two peaks in the vicinity of the oxygen peak. These were identified as mass #32 and mass #34. Upon admitting oxygen to the system, the mass #34 peak increased with a magnitude relative to that of oxygen. Shutting off both the oxygen leak and ion pump produced four peaks, with mass numbers 34, 32, 30 and 28. With the pump off, masses 32 and 34 remained approximately the same as before but masses 30 and 28 were the prominent peaks. Admitting oxygen with the furnace hot, increased masses 32 and 34 with the same relative magnitude. During the remainder of the tests, the double peak effect was not noticed on the "P" partial pressure analyzer.

Run # 2

Run # 2 was a "step" run using oxygen as the contaminating gas with specimen Cb-1Zr #5. Position, inscription location and distance from P_1 and P_3 ion gages are the same as Run #1. The following is the order of the steps used and the approximate values at P_1 ion gage.

1. 1.5(-5) at 1100°C*
2. 1.5(-5) at 980°C
3. 1.5(-5) at 885°C
4. 3.5(-6) at 885°C
5. 3.6(-6) at 980°C
6. 3.6(-6) at 885°C
7. 5(-7) at 1100°C
8. 5(-7) at 980°C
9. 5(-7) at 885°C

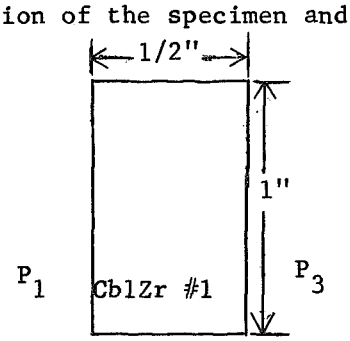
*The numbers in parenthesis are powers of ten.

The second set of ion gages were used for this run and their calibration curves are in Section 11.1 Calibration of Ion Gages. The first set of partial pressure analyzer calibrations and sensitivity values were used for this run and can be found in Section 11.2 Partial Pressure Analyzer Calibrations.

In the early stages of the second run, the "S" PPA which had a lanthanum boride coated rhenium filament developed problems. The lanthanum boride coating appeared to have evaporated off during the high oxygen stages of the run but the "S" PPA remained running on the bare rhenium filament. It was uncertain the effect this has had in the calibration, and this is to be kept in mind when the percent compositions for the "S" PPA are tabulated.

Run #3

Run #3 was a "step" run using oxygen as the contaminating gas with a smaller specimen, Cb-1Zr #1. The smaller sample was used to check pressure drop differences between the smaller specimen and the regular size samples. The following are the position of the specimen and data concerning the run.



The face shown in the sketch is the top of the sample as it was in the quartz tube. The inscription Cb1ZR 1 is on the south edge of the specimen whereas P_1 is on the west edge and P_3 on the east edge. The distance from P_1 ion gage to the front edge of the specimen was 12 inches. The distance from the P_3 ion gage to the back edge of the specimen was 22 3/4 inches. The following is the order of the steps used and approximate values at P_1 ion gage.

1. 1(-5) at 1100°C	4. 4.2(-6) at 885°C	7. 1.7(-6) at 1100°C
2. 1(-5) at 980°C	5. 4.2(-6) at 980°C	8. 1.7(-6) at 980°C
3. 1(-5) at 885°C	6. 4.2(-6) at 1100°C	9. 1.7(-6) at 885°C

The third set of ion gages were used for this run, their calibration curves are in Section 11.1 Calibration of Ion Gages. The second set of partial pressure analyzer calibrations and sensitivity values were used for this run and can be found in Section 11.2 Partial Pressure Analyzer Calibrations. These calibrations were used throughout the remaining nine runs.

One of the problems during the run resulted from the fact that the system was not able to get into the 10^{-7} range. Therefore, 1.7×10^{-6} at P_1 was picked as the third pressure step. It should be noted that the oxygen was shut off on 5/21/67 from 12:22 PM to 2:10 PM. Thus, the running time of the step 1.7×10^{-6} at 1100°C was 25 hours and 17 minutes whereas all the other eight steps were of 24 hours duration.

Run # 4

Run # 4 was a "steady state" run using water vapor as the contaminating gas with regular size specimen, Cb-1Zr # 1. As stated before, the position and distances of the specimen are the same as in Run # 1. The pressure at P_1 was held at approximately 5×10^{-6} torr and specimen temperature at 980°C. In this run, the palladium leak on the system which is used to remove H_2 produced during the dissociation of water vapor on the hot columbium surface, was activated. A five liter/sec Varian ion pump was placed on the other side of the leak to pump the H_2 away. Figure 6.1 shows the position of the palladium leak with respect to the rest of the system.

The run was terminated at 12:10 PM on June 12, 1967 due to a system failure. A crack developed on the P_o ion gage after seven days running time and the

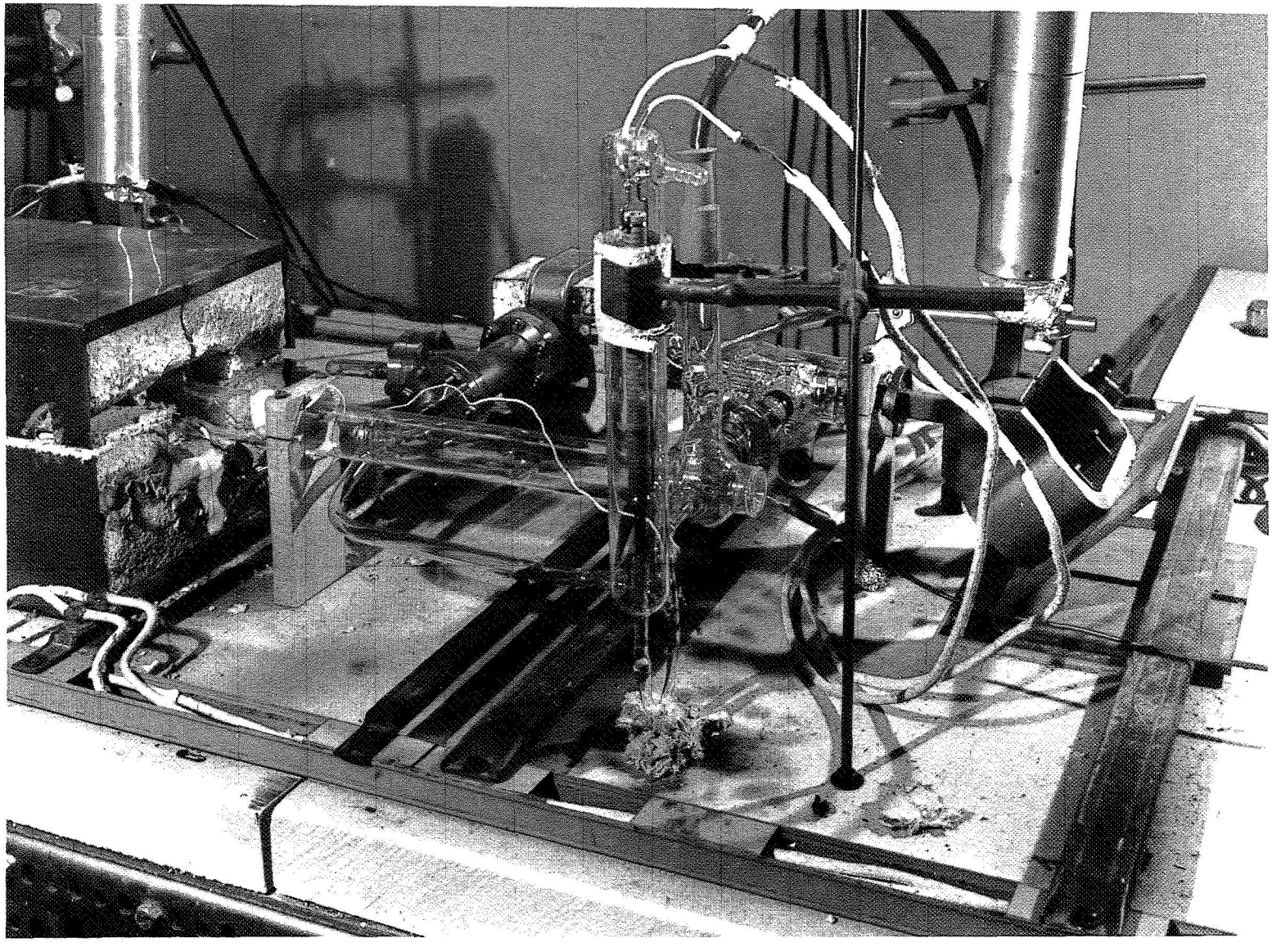


Figure 6.1. Five Liter per Second Varian Ion Pump and Hydrogen Leak

system came up to atmospheric pressure. The sample was at 980°C approximately three minutes during the system failure.

Run # 5

Run # 5 was a "step" run using water vapor as the contaminating gas with specimen Cb-1Zr #2. The following is the order of the steps used and approximate values at the P_1 ion gage.

1. 4(-5) at 1100°C	4. 5(-6) at 885°C	7. 7(-7) at 1100°C
2. 4(-5) at 980°C	5. 5(-6) at 980°C	8. 7(-7) at 980°C
3. 4(-5) at 885°C	6. 5(-6) at 1100°C	9. 7(-7) at 885°C

Run # 6

Run # 6 was a "step" run using water vapor as the contaminating gas with specimen T a #77. The following is the order of the steps used and approximate values at the P_1 ion gage.

1. 5(-5) at 1100°C	4. 4.5(-6) at 885°C	7. 4(-7) at 1100°C
2. 5(-5) at 980°C	5. 4.5(-6) at 980°C	8. 4(-7) at 980°C
3. 5(-5) at 885°C	6. 4.5(-6) at 1100°C	9. 4(-7) at 885°C

Run # 7

Run # 7 was a "step" run using water vapor as the contaminating gas with specimen T-111#84. The following is the order of the steps and approximate values at the P_1 ion gage.

1. 6(-5) at 1100°C	4. 3.5(-6) at 885°C	7. 6.5(-7) at 1100°C
2. 6(-5) at 980°C	5. 3.5(-6) at 980°C	8. 6.5(-7) at 980°C
3. 6(-5) at 885°C	6. 3.5(-6) at 1100°C	9. 6.5(-7) at 885°C

Run # 8

Run # 8 was a "step" run using water vapor as the contaminating gas with specimen Ta-10W #80. The following is the order of the steps used and approximate values at the P_1 ion gage.

1. 7.5(-5) at 1100°C	4. 2.0(-6) at 885°C	7. 5(-7) at 1100°C
2. 7.5(-5) at 980°C	5. 6.0(-6) at 980°C	8. 5(-7) at 980°C
3. 7.5(-5) at 885°C	6. 6.0(-6) at 1100°C	9. 5(-7) at 885°C

Run # 9

Run # 9 was a "step" run using water vapor as the contaminating gas with specimen Ta-$2H_f$ #1. The following is the order of the steps used and approximate values at the P_1 ion gage.

1. 4.5(-5) at 1100°C	4. 2.5(-6) at 885°C	7. 8.5(-7) at 1100°C
2. 4.5(-5) at 980°C	5. 2.5(-6) at 980°C	8. 8.5(-7) at 980°C
3. 4.5(-5) at 885°C	6. 2.5(-6) at 1100°C	9. 8.5(-7) at 885°C

Run # 10

Run # 10 was a "steady state" run using water vapor as the contaminating gas with specimen T-111 #85. The test was run at a pressure of approximately 5×10^{-5} torr at P_1 ion gage and a specimen temperature of 980°C.

Run # 11

Run # 11 was a "steady state" run using carbon monoxide as the contaminating gas with specimen Ta #76. The test was run at a pressure of approximately 6×10^{-6} torr at P_1 ion gage and a specimen temperature of 980°C.

Run # 12

Run # 12 was a "step" run using carbon monoxide as the contaminating gas with specimen Ta #78. The following is the order of the steps used and approximate values at P_1 ion gage.

1. 5.0(-5) at 1100°C	4. 7.5(-6) at 885°C	7. 5.5(-7) at 1100°C
2. 8.5(-5) at 980°C	5. 6.0(-6) at 980°C	8. 4.0(-7) at 980°C
3. 6.5(-5) at 885°C	6. 5.0(-6) at 1100°C	9. 4.0(-7) at 885°C

7.0 DATA REDUCTION

As mentioned in the Introduction, the principal data reduction was accomplished by NASA at the Lewis Research Center. The only data reduction performed by General Electric was the determination of corrected pressure analysis resulting in the determination of sensitivity values for each of the mass spectrometer tubes. The data reduction in general followed that of Barrett(1) with the noted modifications.

8.0 EXPERIMENTAL DATA AND CALCULATIONS

The data were analyzed and reduced to their present form at the NASA - Lewis Research Center, Cleveland, Ohio. A summary of the reduced data is presented in Tables 8.1 and 8.2. Table 8.1 contains the experimental conditions of each run as well as the calculated values of the gettering rate, accumulated weight gain, mass flux and sticking probability. Table 8.2 contains the gas analysis data that was taken throughout the experiment.

8.1 Sample Calculations

The following sample calculations were made on the data obtained during the first time period of run #5 where the specimen was columbium - .89% zirconium and the contaminant was water vapor. The experimental constants are,

$$M = 18 \text{ g/mole}$$

$$T_o = T_3 = 296^{o}K$$

$$a = 21.79 \text{ cm}^2$$

Refer to Section 3 for the remainder of the constants.

8.1.1 Gettering Rate

The gettering rate $\dot{m}$, is the rate per unit area at which the specimen absorbes the contaminant gas. The gettering rate (based on readings taken hourly and then averaged over the test time interval) is calculated as follows:

$$\dot{m} = \frac{MQ_o}{RT_o a} \quad (3600 \times 10^6) \quad \mu g \text{ cm}^{-2} \text{ hr}^{-1}$$

Since,

$$Q_o = \Delta PC_o = \Delta PB \ (T_o/M)^{1/2},$$

Where B is defined in 11.3

Table 8.1. Experimental Data

Specimen	Time hrs	Temperature °K	Corrected Pressures P_0 torr	P_1 torr	P_3 torr	Gettering Rate M $\mu g\ cm^{-2}\ hr^{-1}$	Accumulated Weight Gain over Test Interval $\mu g\ cm^{-2}$	G $\mu g\ cm^{-2}\ hr^{-1}$	Sticking Probability e	Contaminant
Cb 1Zr #7	242.75	1253	3.52(-6)	3.75(-7)	3.26(-8)	0.3693	138.3	2.247	0.2535	O_2
Cb 1Zr #5	23	1373	1.83(-4)	1.77(-5)	2.22(-6)	30.33	697.6	153.1	0.1982	O_2
	24	1253	1.86(-4)	1.78(-5)	2.09(-6)	30.96	743.0	144.5	0.2142	
	23	1158	1.81(-4)	2.18(-5)	4.68(-6)	29.27	763.2	323.3	0.09054	
	22	1158	3.84(-5)	4.02(-6)	9.62(-7)	6.310	138.8	66.41	0.09503	
	23	1253	3.89(-5)	4.08(-6)	8.48(-7)	6.402	147.25	58.55	0.1093	
	23	1373	3.70(-5)	4.23(-6)	9.04(-7)	6.022	138.5	62.36	0.09656	
	20	1373	1.17(-6)	1.55(-7)	3.48(-7)	0.186	3.720	2.398	0.07762	
	23	1253	1.52(-6)	1.21(-7)	3.84(-8)	0.257	5.911	2.649	0.09711	
	22	1158	1.54(-6)	1.23(-7)	3.59(-8)	0.260	5.720	2.475	0.1052	
Cb-0.75 Zr #1 Short sample	24	1373	1.40(-4)	1.08(-5)	2.68(-6)	67.98	1532	184.9	0.3677	O_2
	24	1253	1.76(-4)	1.45(-5)	6.24(-6)	84.75	1954	431.0	0.1966	
	24	1158	1.73(-4)	1.80(-5)	9.20(-6)	81.57	1893	634.6	0.1285	
	24	1158	4.31(-5)	6.37(-6)	2.95(-6)	19.33	452.5	203.8	0.09487	
	24	1253	4.07(-5)	5.38(-6)	2.34(-6)	18.58	435.0	161.5	0.1150	
	24	1373	3.98(-5)	6.72(-6)	1.81(-6)	17.40	430.0	125.0	0.1391	
	25	1373	1.06(-5)	9.72(-7)	4.69(-7)	5.049	113.9	32.34	0.1561	
	24	1253	1.03(-5)	1.24(-6)	6.91(-7)	4.790	102.9	47.66	0.1005	
	24	1158	1.03(-5)	1.43(-6)	8.67(-7)	4.678	100.6	59.82	0.09819	
Cb-1Zr #1	163	1253	1.32(-5)	4.37(-6)	3.70(-6)	1.23	200.5	191.3	6.497(-3)	H_2O
Cb-1Zr #2	23	1373	1.20(-4)	3.71(-5)	3.09(-5)	11.35	263.1	1599	7.053(-3)	H_2O
	23	1253	1.40(-4)	4.08(-5)	3.36(-5)	13.47	313.1	1738	7.727(-3)	
	23	1158	1.30(-4)	4.00(-5)	3.39(-5)	12.30	285.1	1751	7.008(-3)	
	23	1158	1.65(-5)	5.89(-6)	4.94(-6)	1.444	33.30	255.8	5.663(-3)	
	23	1253	1.65(-5)	4.94(-6)	4.00(-6)	1.568	36.14	207.0	7.572(-3)	
	23	1373	1.67(-5)	4.95(-6)	4.07(-6)	1.598	36.88	210.5	7.583(-3)	
	23	1373	1.64(-6)	6.67(-7)	5.62(-7)	0.132	3.072	29.07	4.553(-3)	
	23	1253	1.53(-6)	6.70(-7)	5.71(-7)	0.117	2.710	29.53	3.938(-3)	
	23	1158	1.51(-6)	6.98(-7)	5.97(-7)	0.110	2.540	30.86	3.553(-3)	
Ta#77	23	1373	1.17(-4)	4.82(-5)	3.80(-5)	9.45	217.1	1964	4.910(-3)	H_2O
	24	1253	1.38(-4)	6.15(-5)	5.15(-5)	10.54	243.6	2660	3.934(-3)	
	24	1158	1.55(-4)	6.73(-5)	5.89(-5)	12.14	280.2	3047	3.981(-3)	
	24	1158	9.07(-6)	4.34(-6)	3.61(-6)	0.6519	15.12	186.9	3.544(-3)	
	24	1253	8.69(-6)	4.13(-6)	3.46(-6)	0.6285	14.51	179.1	3.513(-3)	
	24	1373	8.08(-6)	3.78(-6)	3.19(-6)	0.5923	13.64	165.2	3.586(-3)	
	23	1373	1.13(-6)	4.74(-7)	4.03(-7)	0.0909	2.008	20.85	4.443(-3)	
	24	1253	9.19(-7)	3.90(-7)	3.13(-7)	0.0730	1.681	16.21	4.506(-3)	
	24	1158	9.27(-7)	3.91(-7)	3.13(-7)	0.0738	1.703	16.21	4.557(-3)	
T-111 #84	23	1373	1.62(-4)	5.68(-5)	4.58(-5)	13.45	309.0	2371	5.654(-3)	H_2O
	23	1253	1.54(-4)	6.89(-5)	5.37(-5)	10.97	253.4	2776	3.954(-3)	
	23	1158	1.44(-4)	7.55(-5)	6.01(-5)	8.826	203.9	3107	2.830(-3)	
	23	1158	7.61(-6)	3.87(-6)	3.04(-6)	0.4804	11.03	157.2	3.126(-3)	
	23	1253	7.38(-6)	3.35(-6)	2.58(-6)	0.5166	11.88	133.5	3.873(-3)	
	23	1373	7.93(-6)	2.88(-6)	2.08(-6)	0.6484	14.97	107.8	6.223(-3)	
	23	1373	2.12(-6)	7.34(-7)	5.44(-7)	0.1780	4.162	28.12	6.281(-3)	
	23	1253	2.05(-6)	6.88(-7)	5.05(-7)	0.1741	4.008	26.11	6.662(-3)	
	23	1158	2.07(-6)	6.84(-7)	4.10(-7)	0.1777	4.094	26.41	6.721(-3)	
Ta - 10W #80	23	1373	1.69(-4)	7.91(-5)	6.42(-5)	12.21	283.5	3324	3.681(-3)	H_2O
	23	1253	1.62(-4)	8.08(-5)	6.68(-5)	11.01	254.5	3458	3.178(-3)	
	23	1158	1.47(-4)	7.53(-5)	6.03(-5)	9.748	225.0	3120	3.123(-3)	
	23	1158	5.34(-6)	2.69(-6)	2.18(-6)	0.3596	7.366	112.6	4.031(-3)	
	23	1253	1.28(-5)	6.39(-6)	5.22(-6)	0.8672	20.00	270.1	3.272(-3)	
	23	1373	1.04(-5)	5.19(-6)	4.39(-6)	0.7063	16.35	227.1	3.092(-3)	
	23	1373	1.64(-6)	6.21(-7)	5.08(-7)	0.1386	2.996	26.30	6.262(-3)	
	23	1253	1.17(-6)	4.95(-7)	4.19(-7)	0.09127	2.106	21.66	4.211(-3)	
	21	1158	1.11(-6)	4.60(-7)	3.91(-7)	0.08794	1.856	20.11	4.363(-3)	
Ta-2Hf #1	23	1373	1.45(-4)	4.43(-5)	3.47(-5)	14.7	333	1790	8.186(-3)	H_2O
	23	1253	1.37(-4)	4.74(-5)	3.82(-5)	13.1	303	1970	6.621(-3)	
	23	1158	1.38(-4)	5.26(-5)	4.48(-5)	12.5	288	2320	5.389(-3)	
	22	1158	5.95(-6)	2.67(-6)	2.08(-6)	0.479	10.54	108	4.519(-3)	
	23	1253	5.53(-6)	2.34(-6)	1.82(-6)	0.466	10.73	94.2	4.958(-3)	
	23	1373	5.51(-6)	2.02(-6)	1.49(-6)	0.509	11.7	77.0	6.775(-3)	
	23	1373	2.64(-6)	8.01(-7)	6.06(-7)	0.268	5.97	31.3	8.550(-3)	
	23	1253	2.34(-6)	8.04(-7)	6.00(-7)	0.224	5.17	31.0	7.222(-3)	
	23	1158	2.37(-6)	8.20(-7)	5.97(-7)	0.226	5.19	30.9	7.306(-3)	
T-111 #85	243	1253	1.106(-4)	5.025(-5)	4.181(-5)	8.271	2010	2163	3.842(-3)	H_2O
Ta #76	239	1253	9.801(-6)	5.549(-6)	4.469(-6)	0.732	173.0	288.3	2.538(-3)	CO
Ta #78	23	1373	1.11(-4)	3.94(-5)	3.43(-5)	12.33	278.6	2214	5.734(-3)	CO
	23	1253	9.95(-5)	5.92(-5)	4.98(-5)	6.938	158.9	3210	2.175(-3)	
	23	1158	1.01(-4)	5.33(-5)	4.48(-5)	8.224	183.6	2888	2.845(-3)	
	23	1158	9.06(-6)	5.20(-6)	4.45(-6)	0.664	15.37	287.3	2.220(-3)	
	23	1253	7.90(-6)	4.45(-6)	3.75(-6)	0.594	13.69	242.2	2.458(-3)	
	23	1373	6.19(-6)	3.64(-6)	3.05(-6)	0.439	10.05	196.9	2.241(-3)	
	23	1373	5.06(-7)	3.96(-7)	2.54(-7)	0.0190	0.425	16.42	1.142(-3)	
	23	1253	4.32(-7)	2.79(-7)	2.03(-7)	0.0263	0.604	13.07	2.017(-3)	
	23	1158	4.48(-7)	3.01(-7)	2.15(-7)	0.0253	0.581	13.85	1.824(-3)	

Table 8.2. Gas Analysis Data

Specimen	Time hrs	Temp. °K	P_0						P_1						P_3					
			$\%O_2$	%CO	$\%CO_2$	$\%H_2O$	$\%H_2$	%He	$\%O_2$	%CO	$\%CO_2$	$\%H_2O$	$\%H_2$	%He	$\%O_2$	%CO	$\%CO_2$	$\%H_2O$	$\%H_2$	%He
Cb 1Zr #7	242.75	1253	99.35	0.65	--	--	--	--	80.80	5.51	0.90	1.23	9.32	2.27	33.83	23.65	11.15	14.93	15.28	1.20
Cb 1Zr #5	23	1373	100	--	--	--	--	--	100	--	--	--	--	--	72.50	9.39	6.63	7.71	3.12	0.66
	24	1253	100	--					100	--	--	--	--	--	76.32	7.49	7.41	4.54	1.65	2.59
	23	1158	100	--	--	--	--	--	100	--	--	--	--	--	81.97	5.14	12.76	--	0.14	0.14
	22	1158	100	--	--	--	--	--	91.07	5.15	3.37	0.18	0.24	--	69.90	16.15	12.43	1.10	0.43	--
	23	1253	100	--	--	--	--	--	93.08	4.82	2.00	--	0.09	--	65.95	17.88	13.25	2.25	0.67	--
	23	1373	100	--	--	--	--	--	92.61	4.46	2.51	--	0.42	--	63.01	16.77	14.77	3.59	1.20	0.66
	20	1373	81.92	7.86	7.53	2.25	0.44	--	27.98	50.24	3.75	2.52	13.87		10.78	62.71	10.94	5.11	10.21	0.25
	23	1253	92.94	5.27	--	1.70	0.08	--	29.56	63.53	--	2.05	3.59		15.16	74.78	0.86	5.55	3.65	--
	22	1158	93.26	5.13	--	1.60	--	--	31.07	66.48	--	1.31	1.14	--	14.73	76.50	--	6.11	2.66	--
Cb - 0.75Zr #1	24	1373	100	--	--	--	--	--	74.65	4.99	5.35	14.08	0.94	--	46.05	17.10	15.23	26.36	2.15	--
	24	1253	100	--	--	--	--	--	82.89	4.38	4.80	7.77	0.16	--	68.86	7.50	14.57	8.66	0.41	--
	24	1158	100	--	--	--	--	--	88.25	3.52	2.44	5.74	0.04	--	78.35	5.04	9.39	6.03	0.39	--
	24	1158	98.76	1.24	--	--	--	--	86.54	8.99	0.10	4.26	0.11	--	66.07	14.00	8.79	9.96	1.15	--
	24	1253	98.50	1.50	--	--	--	--	85.26	9.48	--	5.19	0.07	--	64.56	15.48	9.13	10.03	0.80	--
	24	1373	98.75	1.25	--	--	--	--	(85.26)	(9.48)	--	(5.19)	(0.07)	--	61.46	20.01	10.99	7.54	--	--
	25	1373	95.46	4.54	--	--	--	--	52.95	30.34	4.42	10.69	1.60	--	39.96	47.95	9.06	6.59	1.41	--
	24	1253	94.75	5.25	--	--	--	--	57.61	29.00	3.37	9.23	0.80	--	48.41	43.82	3.00	4.08	0.69	--
	24	1158	94.89	5.11	--	--	--	--	61.89	27.44	2.77	7.37	0.52	--	53.01	35.82	5.22	5.06	0.88	--
Cb - 1Zr #1	163	1253	6.49	1.12	0.02	89.49	2.70	0.22	--	0.30	--	13.40	85.93	0.37	--	4.60	0.15	30.02	61.09	4.15
Cb - 1Zr #2	23	1373	4.09	0.13	--	93.00	2.53	0.25	--	0.02	--	6.40	93.44	0.14	--	1.20	--	24.90	69.27	4.63
	23	1253	4.64	--	--	93.93	1.21	0.22	--	0.16	--	8.94	90.79	2.50	--	0.59	--	44.76	52.15	2.50
	23	1158	4.84	--	--	94.05	1.11	--	--	--	--	13.18	86.80	0.02	--	0.64	--	65.89	32.19	1.28
	23	1158	5.87	0.44	--	92.81	0.86	--	0.11	1.28	--	46.30	52.27	0.05	0.44	2.58	0.13	66.26	30.18	0.41
	23	1253	5.73	0.26	--	93.21	0.79	--	--	0.08	--	18.12	81.79	0.01	0.09	1.83	--	20.71	76.72	0.64
	23	1373	5.60	0.26	--	9325	0.88	--	--	0.05	--	16.77	83.18	--	0.08	2.95	0.16	17.17	78.77	0.88
	23	1373	6.38	2.10	--	87.00	4.32	--	--	7.18	--	28.87	63.78	0.17	0.52	10.38	0.72	9.44	78.01	0.93
	23	1253	6.07	2.92	--	84.66	6.36	--	--	11.11	--	31.06	57.77	0.07	0.54	13.13	0.69	5.43	79.53	0.69
	23	1158	6.33	3.59	--	85.25	4.82	--	--	13.06	--	31.76	55.11	0.08	0.78	14.46	0.70	6.26	77.16	0.65
Ta #77	23	1373	1.42	--	--	93.42	5.15	--	--	--	--	44.31	55.69	--	0.92	0.41	0.06	93.12	5.18	--
	24	1253	5.27	--	--	88.71	6.03	--	--	--	--	53.06	46.94	--	--	0.49	--	98.37	1.13	--
	24	1158	5.54	--	--	92.08	2.32	--	0.25	--	--	83.01	16.74	--	--	--	--	100.00	--	--
	24	1158	4.38	--	--	92.31	3.31	--	0.45	--	--	69.06	30.49	--	1.06	--	--	97.87	1.07	--
	24	1253	4.47	--	--	86.68	8.85	--	0.47	--	--	75.17	24.36	--	1.70	0.01	0.02	97.03	1.24	--
	24	1373	4.63	--	--	91.75	3.62	--	0.90	--	--	93.67	5.43	--	1.52	--	--	96.78	1.69	--
	23	1373	8.65	--	--	83.94	7.40	--	2.09	--	--	40.13	57.78	--	6.30	1.58	--	79.36	12.76	--
	29	1253	9.41	--	--	82.53	8.06	--	3.49	--	--	69.35	27.15	--	9.14	1.32	--	71.79	17.75	--
	29	1158	10.83	--	--	81.71	7.46	--	8.41	--	--	17.37	74.22	--	9.01	1.11	0.58	69.60	19.70	--

( ) Estimated values used when no values are available

Table 8.2 (continued). Gas Analysis Data

T - 111 #84	23	1373	1.06	--	--	90.49	8.45	--	--	--	--	56.07	43.91	0.02	0.85	0.39	1.47	85.35	**11.00**	0.94
	23	1253	--	--	--	91.93	8.07	--	--	--	--	100	--	--	--	--	--	98.06	1.94	--
	23	1158	3.96	--	--	94.76	1.27	--	--	--	--	66.72	33.28	--	0.50	--	--	18.42	1.08	--
	23	1158	3.93	--	--	94.82	1.24	--	--	--	--	49.77	50.23	--	0.27	--	0.04	95.40	3.78	--
	23	1253	4.03	--	--	94.64	1.33	--	--	--	--	57.71	42.29	--	--	--	1.67	93.73	4.60	--
	23	1373	3.63	--	--	95.44	0.93	--	--	--	--	37.50	62.50	--	0.39	0.04	0.78	67.32	31.47	--
	23	1373	4.81	--	--	88.92	6.27	--	--	--	--	17.65	82.35	--	0.59	0.13	--	13.34	85.88	--
	23	1253	4.09	--	--	89.45	6.46	--	--	--	--	22.99	77.01	--	0.51	0.09	--	10.07	89.32	--
	23	1158	3.93	--	--	89.89	6.18	--	--	--	--	25.58	74.42	--	2.73	0.30	--	55.20	41.77	--
Ta -10W #80	23	1373	--	--	--	95.67	4.33	--	--	--	--	63.24	36.76	--	2.86	--	0.97	93.36	2.81	--
	23	1253	4.77	0.74	--	93.33	1.16	--	--	--	--	70.54	29.46	--	2.28	--	1.14	93.40	3.18	--
	23	1158	4.66	0.32	--	94.44	0.58	--	--	--	--	66.36	33.64	--	1.17	0.29	--	95.44	3.10	--
	23	1158	4.28	0.82	--	93.82	1.08	--	--	--	--	39.22	60.78	--	2.73	0.79	--	89.96	9.53	--
	23	1253	7.56	0.80	--	90.38	1.27	--	--	--	--	99.39	0.61	--	1.32	--	1.50	92.81	4.37	--
	23	1373	4.90	0.50	--	93.72	0.89	--	--	--	--	66.58	33.42	--	0.99	0.62	--	92.62	5.77	--
	23	1373	10.36	1.22	1.12	82.77	4.52	--	2.60	--	0.29	48.63	48.48	--	4.05	2.10	3.26	58.81	31.78	--
	23	1253	11.76	1.28	0.35	82.12	4.49	--	0.63	--	1.61	40.86	56.89	--	4.99	2.05	1.74	52.29	38.70	0.23
	21	1158	12.07	1.23	0.20	82.97	3.54	--	0.71	--	--	39.57	59.72	--	5.43	2.07	1.21	48.78	42.35	0.16
Ta-2Hf #1	23	1373	34.85	0.25	--	51.98	12.92	--	0.89	--	--	36.03	63.04	0.04	0.32	1.97	0.40	86.65	8.80	1.85
	23	1253	20.25	--	--	27.26	52.49	--	--	--	--	55.73	44.24	0.02	0.61	0.74	0.36	91.87	5.84	0.58
	23	1158	4.48	--	--	89.19	6.33	--	--	--	--	63.61	36.39	--	0.69	0.22	0.25	92.04	6.42	0.37
	22	1158	--	--	--	(92.33)	(3.47)	--	--	--	--	(39.71)	(60.29)	--	(1.64)	(0.17)	(--)	(85.42)	(12.73)	(--)
	23	1253	3.89	--	--	92.83	3.27	--	--	--	--	39.71	60.29	--	1.64	0.17	--	85.42	12.73	--
	23	1373	3.67	--	--	93.08	3.25	--	2.70	--	0.07	78.61	18.54	0.04	1.72	1.42	0.74	66.72	28.82	0.59
	23	1373	4.18	0.51	--	88.98	6.33	--	1.85	--	--	20.10	78.03	0.03	1.10	2.85	0.81	17.25	77.55	0.44
	23	1253	4.42	0.60	--	89.10	5.89	--	1.74	--	--	23.58	74.68	--	0.97	2.14	0.44	15.53	80.69	0.24
	23	1158	4.46	0.66	--	89.20	5.69	--	1.48	--	--	28.28	70.25	--	0.86	1.38	0.86	12.61	84.66	0.20
T - 111 #81	243	1253	4.13	--	--	92.05	3.83	--	--	--	--	92.20	7.80	--	0.49	0.05	0.08	92.29	6.81	0.29
Ta #76	239	1253	0.16	99.60	0.24	--	--	--	--	97.90	1.98	--	0.12	--	--	--	--	--	--	--
Ta #78	23	1373	--	99.71	0.29	--	--	--	1.20	98.80	--	--	--	--	--	97.40	2.60	--	--	--
	23	1253	--	97.02	2.78	--	--	--	--	100.00	--	--	--	--	--	97.44	2.56	--	--	--
	23	1158	--	99.60	0.40	--	--	--	--	100.00	--	--	--	--	--	98.34	1.66	--	--	--
	23	1158	--	100.00	--	--	--	--	--	100.00	--	--	--	--	--	93.53	6.47	--	--	--
	23	1253	--	100.00	--	--	--	--	--	100.00	--	--	--	--	--	95.19	4.81	--	--	--
	23	1373	--	100.00	--	--	--	--	--	100.00	--	--	--	--	--	96.56	3.44	--	--	--
	23	1373	--	100.00	--	--	--	--	--	100.00	--	--	--	--	--	96.21	3.79	--	--	--
	23	1253	--	100.00	--	--	--	--	--	100.00	--	--	--	--	--	95.40	4.60	--	--	--
	23	1158	--	100.00	--	--	--	--	--	100.00	--	--	--	--	--	97.68	2.32	--	--	--

( ) Estimated values used when no values are available

then

$$\dot{m} = (P_o - P_1)\ (M/T_o)\ B\ (T_o/M)^{1/2}\ (3600 \times 10^6/Ra)\ \mu g cm^{-2} hr^{-1} \qquad (8.1)$$

Substituting the appropriate values into equation 7.1 yields

$$\dot{m} = (8.29 \times 10^{-5})\ (18/296)(.203)(296/18)^{1/2}(3600 \times 10^6/62.3 \times 21.79)$$

$$\dot{m} = 11.3\ \mu g\ cm^{-2}\ hr^{-1}$$

Based on calculations for hourly readings the coefficient of variation over the test interval was generally 10% for a given value.

8.1.2 Accumulated Weight Gain

The accumulated weight gain per unit area of the specimen during the duration of a test at one set of conditions is based on successive hourly summing of the rate at each hour.

$$W_p/a = \sum_{i=o}^{i=n-1} \frac{\dot{m}_i + \dot{m}_{i+1}}{2} \Delta t \quad \mu g/cm^2 \qquad (8.2)$$

8.1.3 Mass Flux

The mass flux, based on the average pressure at P_3, may be calculated by the following equation.

$$G = 209.88 \times 10^6\ \bar{P}_3\ (M/T_3)^{1/2} \quad \mu g\ cm^{-2}\ hr^{-1} \qquad (8.3)$$

Substituting the appropriate values into equation 8.3 yields

$$G = 209.88 \times 10^6\ (3.09 \times 10^5)\ (18/296)^{1/2}$$

$$G = 1590\ \mu g\ cm^{-2}\ hr^{-1}$$

8.1.4 Sticking Probability

The sticking probability E, is that fraction of molecules that impinge upon the specimen's surface and that actually stick and react with the metal. The sticking probability is the gettering rate divided by the mass flux.

$$E = \dot{m}/G$$

$$E = 11.3/1590 = 7.1 \times 10^{-3} \qquad (8.4)$$

8.1.5 Differential Weight Gains

The weight gain of a specimen during a test run was determined by three methods. These methods are gravimetric analysis, gas analysis and the pressure drop method described in this report. These weight gains are designated ΔWg, ΔWc and ΔWp respectively.

The total weight gain ΔWp is determined by summing all of the accumulated weight gains during a test and multiplying by the sample's surface area. For run #2 with samples Cb-1Zr#5,

$$\Delta Wp = 0.00255377 \text{ gcm}^{-2} \times 21.4636 \text{ cm}^2 = .05481 \text{ g}$$

9.0 RESULTS AND DISCUSSION

A total of twelve metal specimens were run during the performances of this contract. Each specimen was exposed to one of three contaminant gases. These gases were oxygen, water vapor and carbon monoxide. Refer to Table 6.1 for the exact sequence of specimen alloys and contaminant used for each test run.

The twelve test runs can be conveniently divided into four groups that are based on the metal composition and the contaminant. These groups are,

I. Nominal Columbium - 1% Zirconium with oxygen

II. Nominal Columbium - 1% Zirconium with water vapor

III. Tantalum and Tantalum alloys with water vapor

IV. Tantalum with carbon monoxide

Each group will be considered separately because of the different properties of the metal specimens and contaminant gases.

9.1 Regression Analysis

It has been demonstrated by Barrett (1), that if log $\dot{m}$ is plotted versus log G the points fall on a nominally straight line. The equation of this line takes the form

$$y = \log \dot{m} = b_1 + b_2 \log G \pm \sigma_y \qquad (9.1)$$

were b_1 and b_2 are constants and σ_y is the standard error of estimate. Barrett has shown that for the case where the metal is columbium or a dilute columbium zirconium alloy and the contaminant is oxygen, b_2 is unity and b_1 is a measure of the sticking probability. This type of regression analysis was conducted on each of the four groups of data.

Table 9.1 lists the calculated values for the constant in the regression equation as well as the calculated values of the sticking probability, the standard error of estimate with its upper and lower limits and the percent of explained variation, R^2. The calculations were made by two methods. The first method uses the regression equation (9.1) directly and determines the values of b_1 and b_2. This is described as the case where b_2 floats. The second method forces the value of b_2 to be exactly one and the remainder of the constants are calculated on this basis. This is the case where b_2 is forced. When b_2 is forced to unity, these will generally be a small additional error that must be absorbed in the other constant. However, much of the data reported in the literature are based upon the forced calculation so this method was used to be consistent. In Figures 9.1 through 9.7 the plot represents the data calculated under the forced condition. The data in these figures are plotted with a solid line, the upper and lower σ_y limits are dashed lines.

Group I. Columbium 1% Zirconium with Oxygen

The regression analysis for the group I materials would be expected to closely approximate the results of Barrett. A plot of log $\dot{m}$ versus log G for runs 1, 2 and 3 is shown in Figure 9.1. There is a certain amount of scatter evident especially at higher gettering rates and flux values. The equation for the best straight line between these points takes the form

$$\log \dot{m} = -.888 + 1.000 \log G \pm .187$$

The values of the constants b_1 and b_2 agree quite favorably with these found by Barrett which are -1.08 and 1.00 respectively. The points as plotted show no significant specimen temperature dependence which is consistent with the results of Barrett[1].

Table 9.1 Results of Regression Calculations

Group	Run	b_2 Case	b_1	b_2	σy	R^2	E	σy^+	σy^-
I	1,2,3	Floating	-0.696	1.045	0.189	0.952			
		Forced	-0.888	1.000	0.187 *	0.951	0.1294	0.199	.0841
II	4,5	Floating	-1.697	1.145	0.0759	0.993			
		Forced	-2.229	1.000	0.126	0.977	5.907(-3)	7.894(-3)	4.421(-3)
III	6	Floating	-2.431	0.989	0.0556	0.997			
		Forced	-2.392	1.000	0.0529 *	0.997	4.057(-3)	4.583(-3)	3.591(-3)
III	7,10	Floating	-2.694	0.898	0.121	0.980			
		Forced	-2.328	1.000	0.146	0.967	4.695(-3)	6.571(-3)	3.355(-3)
III	8	Floating	-2.653	0.941	0.0650	0.995			
		Forced	-2.438	1.000	0.0829	0.991	3.644(-3)	4.410(-3)	3.010(-3)
III	9	Floating	-2.244	0.986	0.105	0.985			
		Forced	-2.190	1.000	0.0986	0.985	6.450(-3)	8.093(-3)	5.140(-3)
IV	11,12	Floating	-2.228	1.117	0.146	0.980			
		Forced	-2.633	1.000	0.171	0.969	2.328(-3)	3.449(-3)	1.542(-3)

*The error is reduced slightly when b_1 is forced to 1.00 since the additional degree of freedom picked up offsets the slightly poorer fit.

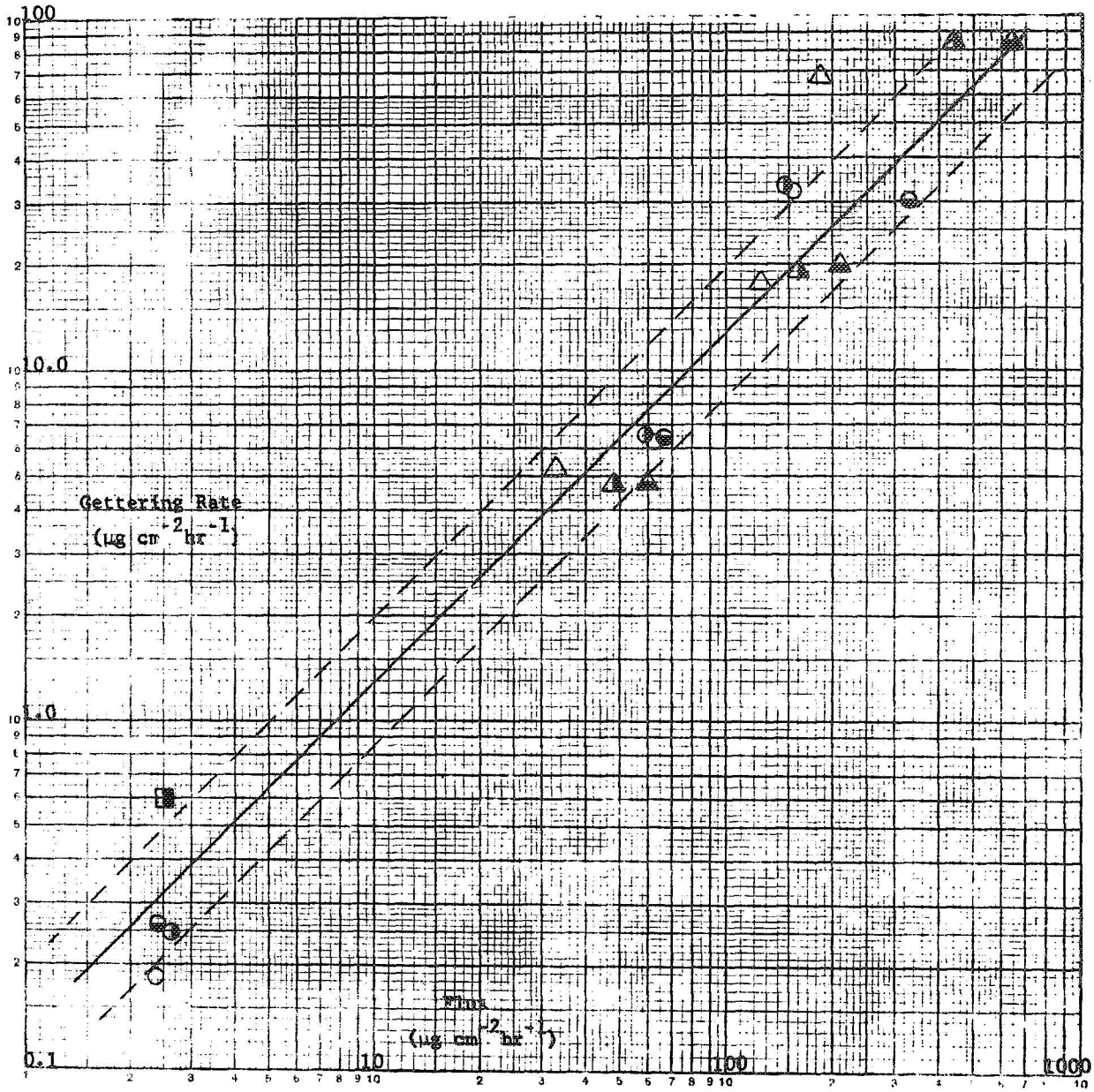


Figure 9.1 Flux versus Gettering Rate for Oxygen on Columbium-Zirconium Alloys. Corrected for Residual Gases.

□ 1-Cb-1Zr-7

○ 2-Cb-1Zr-5

△ 3-Cb-.75Zr-1

Open: 1373°K
Right half shaded: 1253°K
Lower half shaded: 1158°K
Dashed Lines: Upper and lower σ_y Boundries

Group II Columbium - 1% Zirconium with Water Vapor

The plot of log $\dot{m}$ versus log G for runs 4 and 5 is shown in Figure 9.2. The equation of the best straight line between these points takes the form

$$\log \dot{m} = -2.229 + 1.000 \log G \pm .126$$

As in group I, the constant b_2 is close to unity. The value of the constant b_1 of -2.229 represents the lower sticking probability of water vapor on columbium zirconium alloys when compared with oxygen on the same alloy. The value of b_1 is actually the value of log $\dot{m}$ that occurs when G is 1.00 $\mu g\ cm^{-2}\ hr^{-1}$. The value of E calculated from b_1 is 5.407×10^{-3} which is within the range of calculated values in Table 8.1.

Group III Tantalum and Tantalum Alloys with Water Vapor

The tantalum specimens that were run with water vapor as the contaminant consisted of a pure tantalum sample and four tantalum alloys. The analysis of these specimens is given in Table 5.1.

The plots of log $\dot{m}$ versus log G for these specimens are presented in Figures 9.3 through 9.6. The plots for both T-111 samples are included in Figure 9.4. There is no apparent temperature effect noticeable.

The derived E values for tantalum alloys are in the order Ta-2Hf > T-111 > Ta-10W. This is in the order predicted prior to test where a getter such as Hf (or Zr) enhances the gettering rate while an addition like W which tends to lower oxygen solubility in Ta (or Cb) would tend to lower it.

Again, there is no obvious temperature dependence.

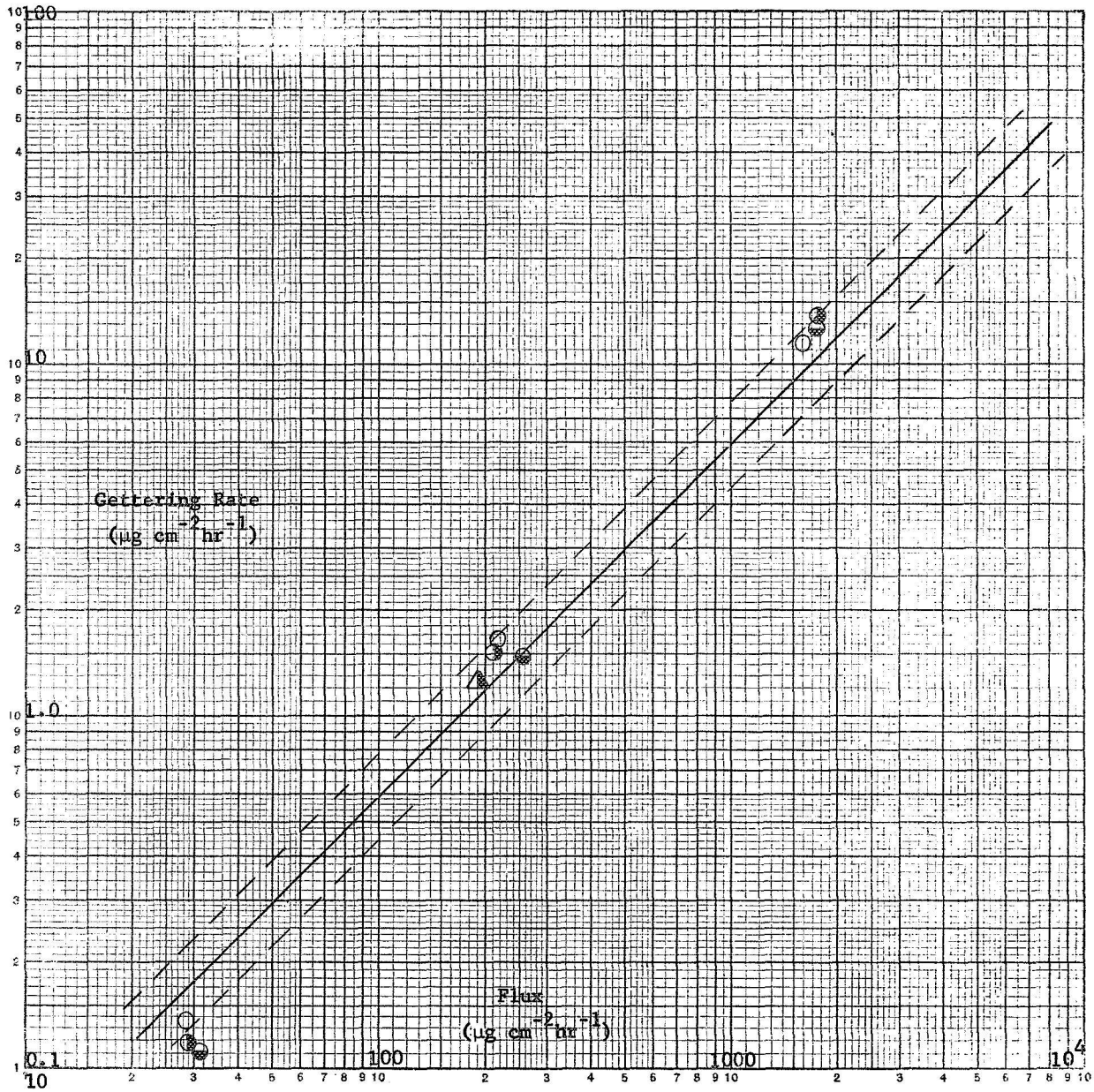


Figure 9.2 Flux Versus Gettering Rate for Water Vapor on Columbium-Zirconium Alloys. Not Corrected for Residual Gases.

△ 4-Cb-1Zr-1

○ 5-Cb-1Zr-2

Open: 1373°K
Right Half Shaded: 1253°K
Lower Half Shaded: 1158°K
Dashed Lines: Upper and Lower σ_y Boundries

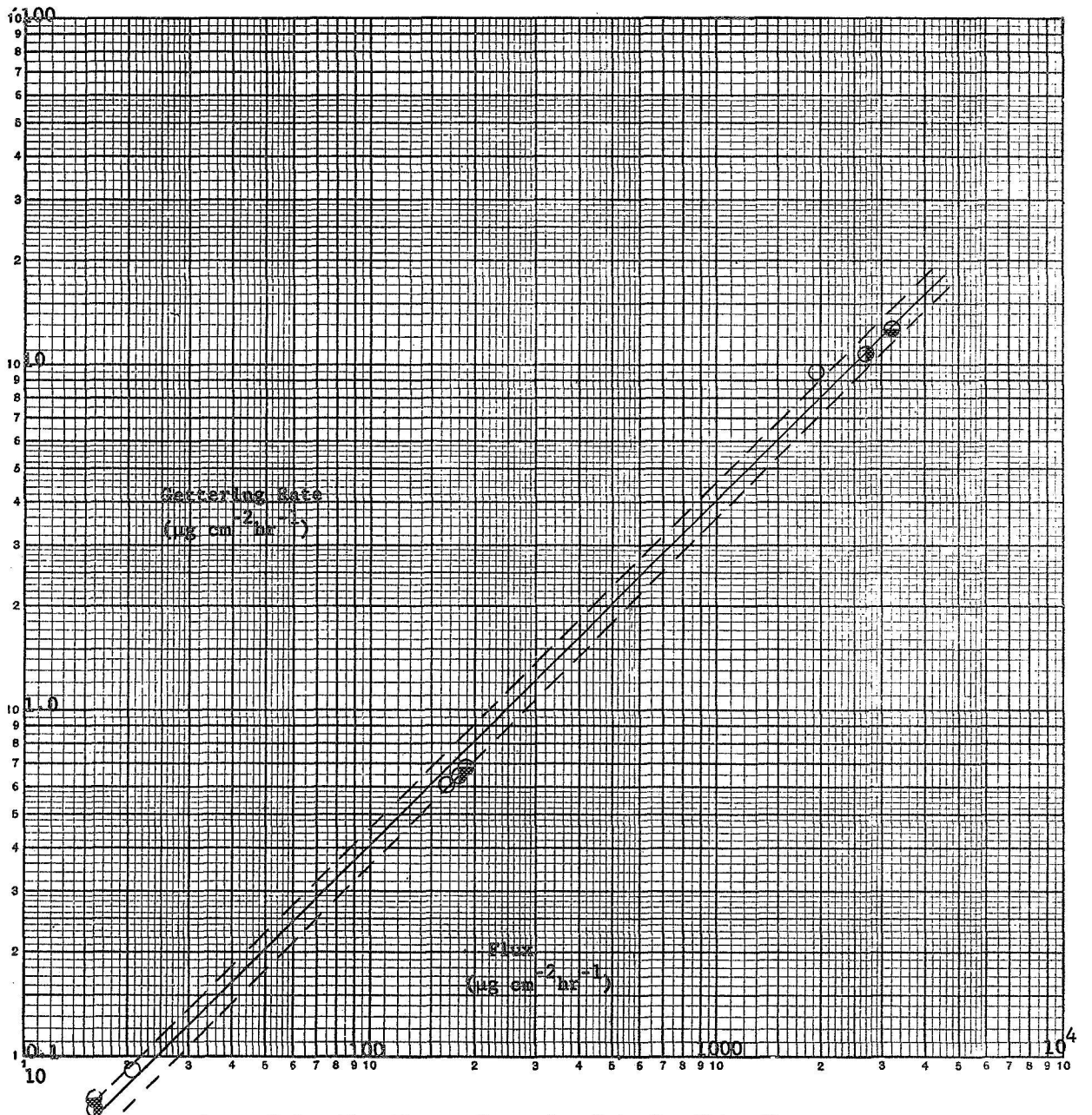


Figure 9.3 Flux Versus Gettering Rate for Water Vapor on Tantalum. Not Corrected For Residual Gases

○ 6-Ta-77

Open: 1373°K
Right Half Shaded: 1253°K
Lower Half Shaded: 1158°K
Dashed Lines: Upper and Lower σ_y Boundries

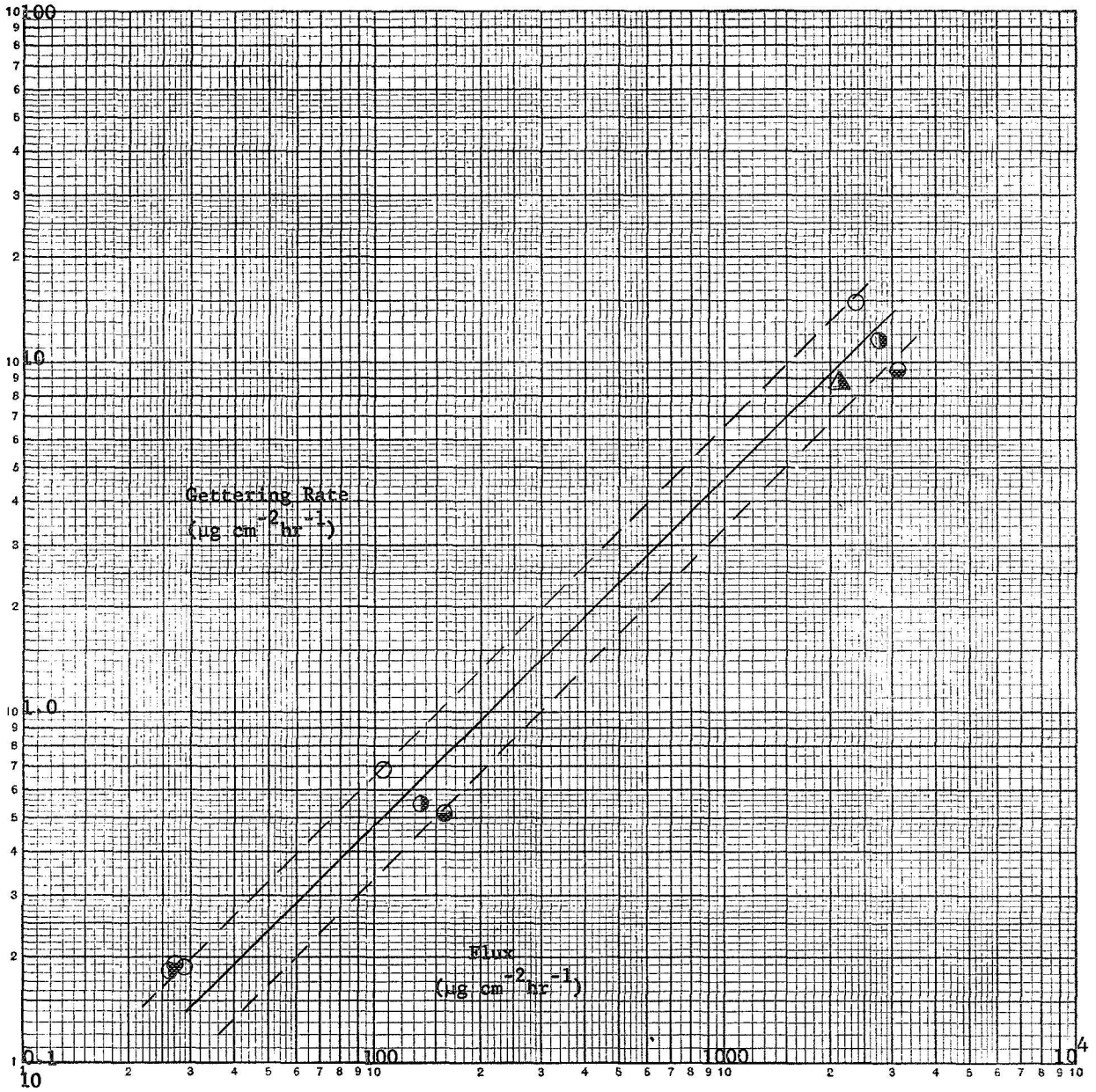


Figure 9.4 Flux Versus Gettering Rate for Water Vapor on Tantalum Alloys. Not Corrected for Residual Gases.

○ 7-T-111-84

△ 10-T-111-85

Open: 1373°K
Right Half Shaded: 1253°K
Lower Half Shaded: 1158°K
Dashed Lines: Upper and Lower σ_y Boundries

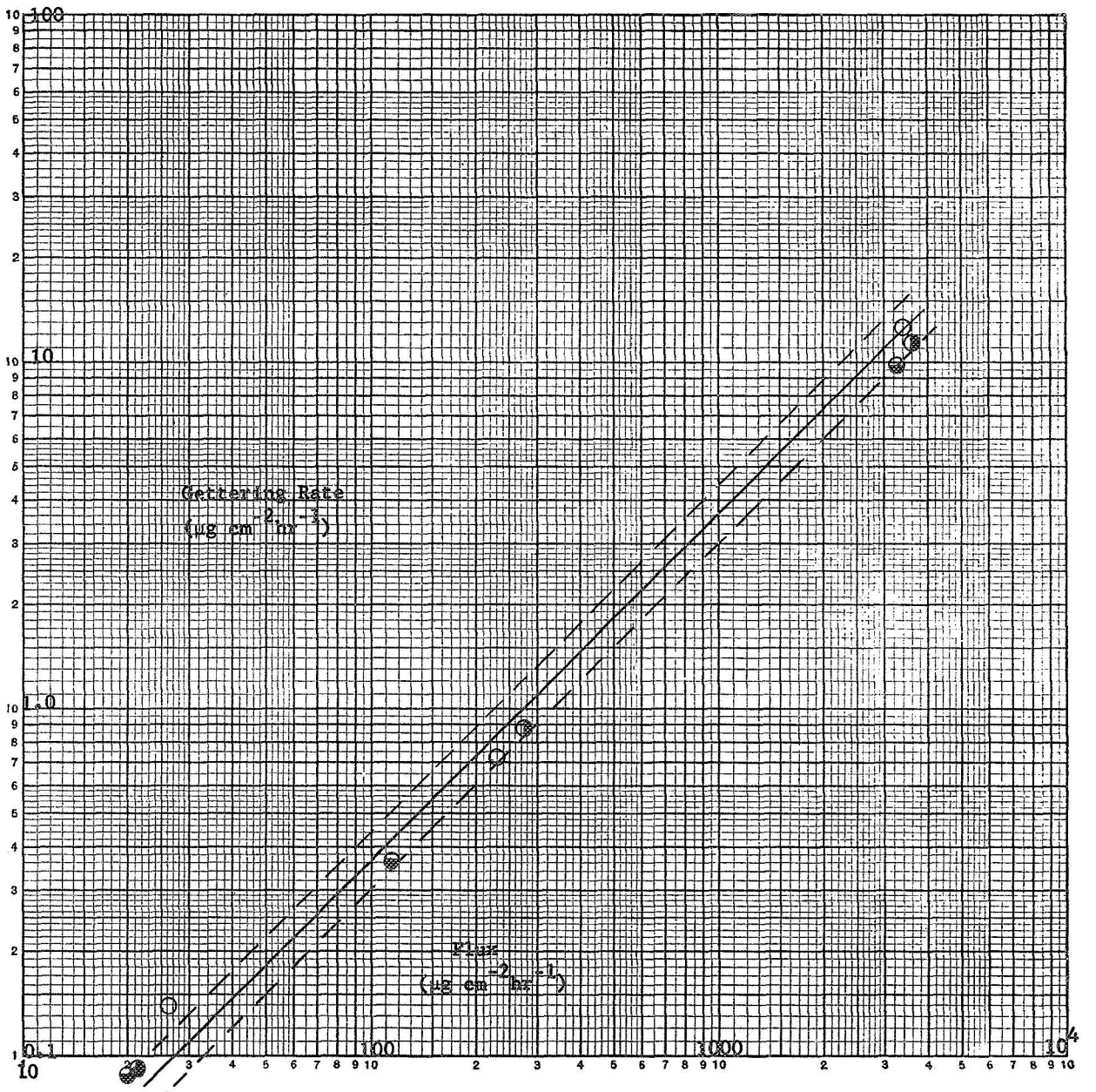


Figure 9.5 Flux Versus Gettering Rate for Water Vapor on Tantalum - 10% Tungsten. Not Corrected for Residual Gases.

○ 8-Ta-10W-80 Open: 1373°K
Right Half Shaded: 1253°K
Lower Half Shaded: 1158°K
Dashed Lines: Upper and Lower σ_y Boundries.

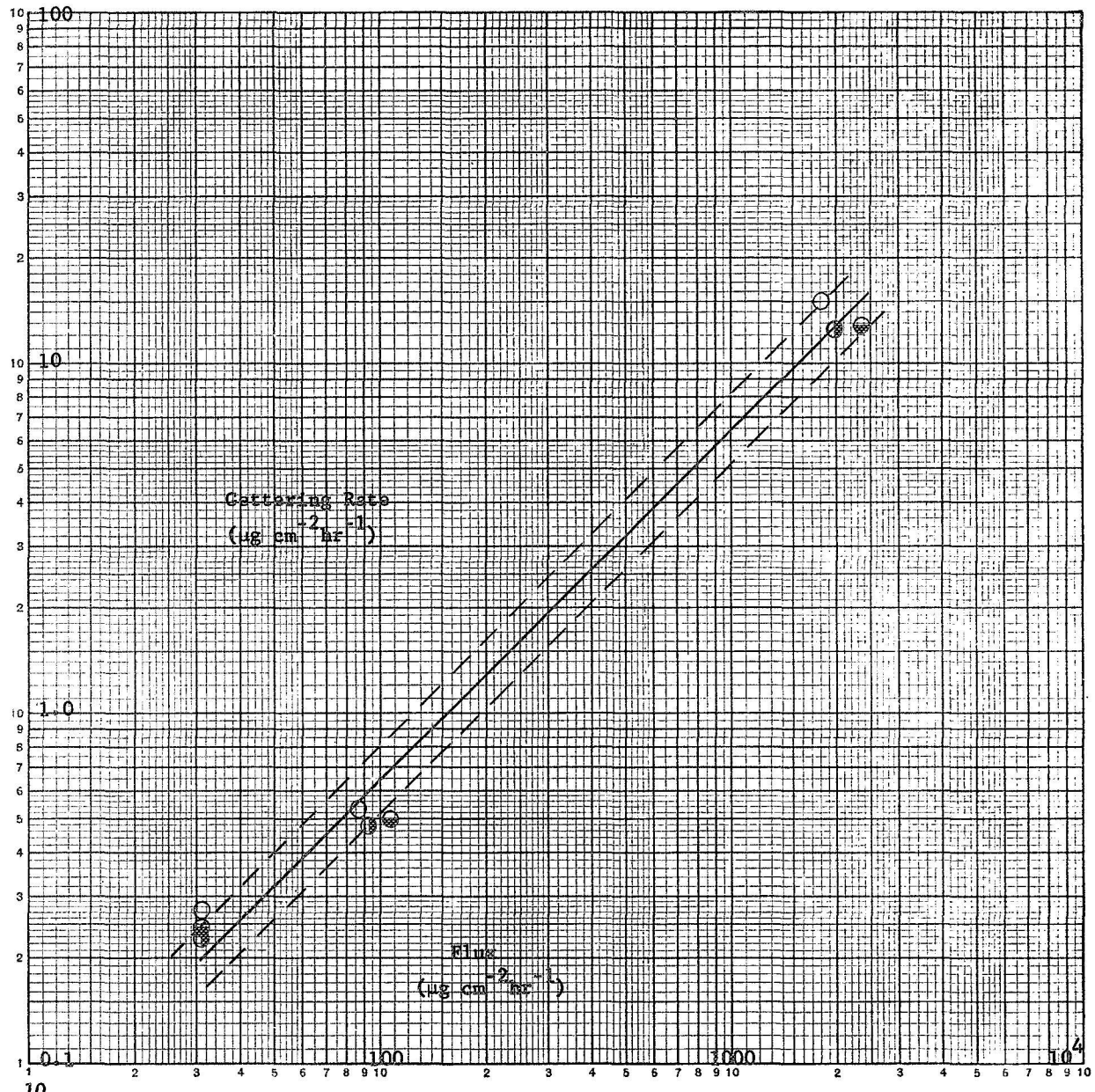


Figure 9.6 Flux Versus Gettering Rate for Water Vapor on Tantalum - 2% Hafnium. Not Corrected for Residual Gases.

○ 9-Ta-2Hf-1

Open: 1373°K
Right Half Shaded: 1253°K
Lower Half Shaded: 1158°K
Dashed Lines: Upper and Lower σ_y Boundries.

Group IV Tantalum with Carbon Monoxide

The plot of log $\mathring{m}$ versus log G for the group IV materials is shown in Figure 9.7. The constants b_1 and b_2 appear to be -2.633 and 1.000 respectively. The sticking probability derived from the intercept is 2.328×10^{-3}. Here also, there is no apparent temperature effect. A sensitivity value of 1.4 was used for these calculations for carbon monoxide.

9.2 Gage Corrections

An ion vacuum gage does not function with equal sensitivity for all of the gases that are involved in this experiment. In general, if two gases at the same true pressure are exposed to a single ion vacuum gage, the gage will not indicate the same true pressure for either gas unless, of course, the gage happens to have been calibrated for that gas. This may also be true for mass spectrometer tubes as well. To correct for this difference, a sensitivity factor is introduced which is primarily a function of the gas but would also be a function of gage type if two gages with different filament materials were compared. Once the sensitivities for the different gases are known, then the proper gage corrections can be made.

In this type of experiment, there is generally a problem with residual gases in the system. During all of the runs except numbers 11 and 12, the contaminant gas decomposes or reacts with material in the vacuum system and results in a build up of undesirable residual gas. The concentrations of these residuals are not small and can contribute a definite error in the pressure measurements. This is especially true at the P_1 and P_3 gage sites that are closes to the specimen. Where the contaminant gas is oxygen, the residual gases are generally carbon monoxide and carbon dioxide. They are formed by the oxidation of residual carbon in the specimen and on the hot ion gage and

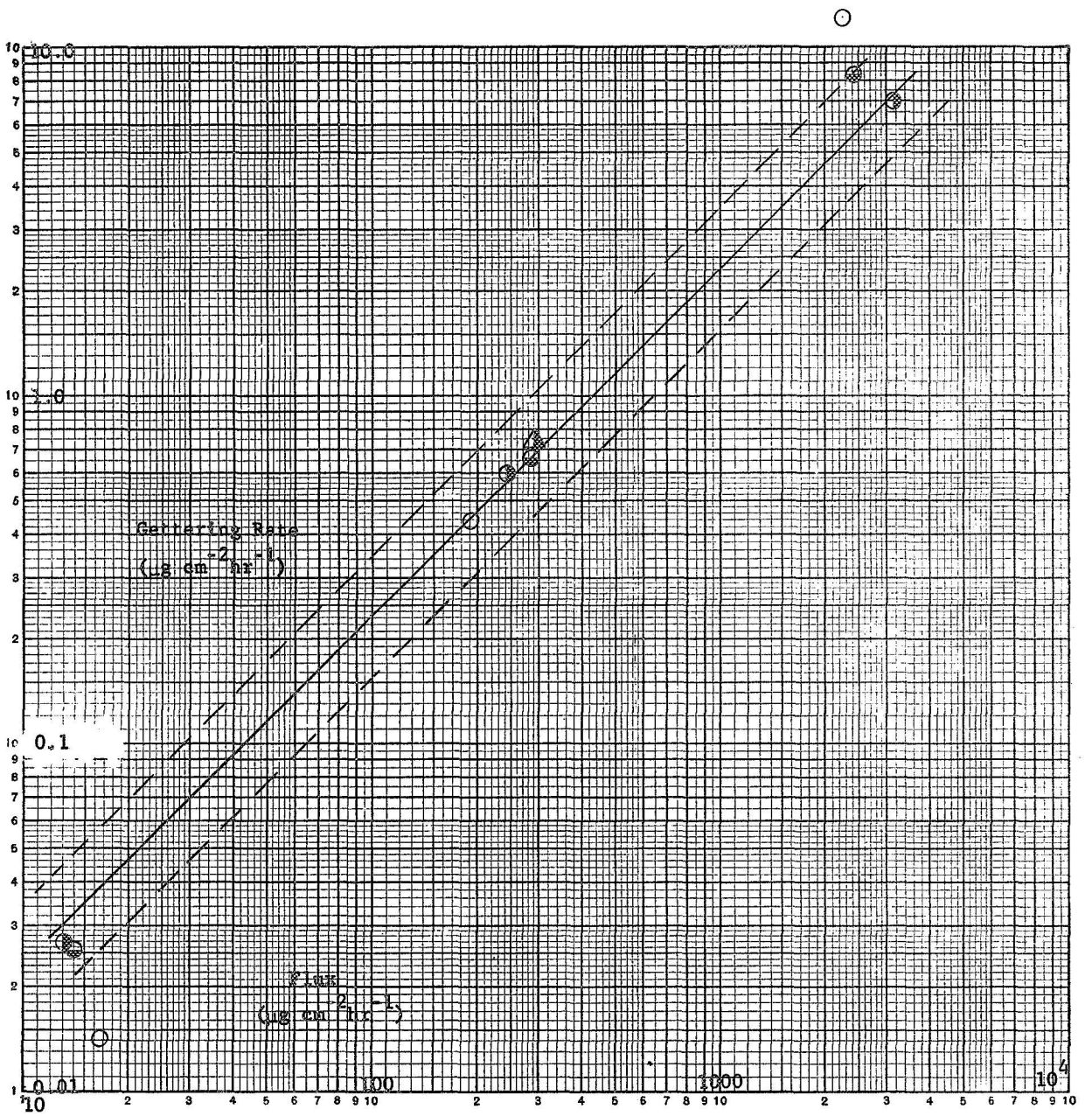


Figure 9.7 Flux Versus Gettering Rate for Carbon Monoxide on Tantalum. Not Corrected for Residual Gases

△ 11-Ta-76

○ 12-Ta-78

Open: 1373°K
Right Half Shaded: 1253°K
Lower Half Shaded: 1158°K
Dashed Lines: Upper and Lower σ_y Boundries

spectrometer filaments. Where the contaminant is water vapor, the residual gases are hydrogen and carbon monoxide. The hydrogen is formed as the water vapor decomposes and the carbon monoxide is formed by the oxidation of carbon as mentioned previously. When carbon monoxide as the contaminant reacts on the speciman surface both the oxygen and carbon are absorbed so no gaseous reaction product is formed.

The gage sensitivity constants K_{O_2}, K_{CO}, K_{CO_2} etc. can be calculated as in the case of oxygen estimated from data taken during the "steady state" runs for each contaminent gas, or they may be found in the literature. Once these values are known, the residual gas correction may be applied.

The total true pressure of a system at equalibrium is the sum of the partial pressures of each component of the system.

$$P_T = P_{O_2} + P_{CO} + P_{CO_2} + P_{H_2O} + P_{H_2} + P_{HE}$$

The total observed pressure at a particular gage is sum of the sensitivity corrected partial pressures of each components,

$$P_{OB} = K_1P_{O_2} + K_2P_{CO_2} + K_3P_{CO} + K_4P_{H_2O} + K_5P_{H_2} + K_6P_{He}$$

where K_1, K_2, K_3 etc. are the sensitivities for each gas involved.

Since

$$P_{O_2}/P_T = \%\ O_2/100$$

and

$$P_{CO}/P_T = \%\ CO/100 \quad \text{etc,}$$

then

$$P_{OB} = K_1 \frac{\%O_2}{100} P_T + K_2 \frac{\%CO}{100} P_T + K_3 \frac{\%CO_2}{100} P_T + K_4 \frac{\%H_2O}{100} P_T + K_5 \frac{\%H_2}{100} P_T + K_6 \frac{\%He}{100} P_T$$

The value of the true pressure P_T can be calculated from this last equation. The partial pressure of each gas in the system can then be computed by rearranging the equation found above,

$$P_{O_2} = P_T \frac{\%O_2}{100}$$

$$P_{CO} = P_T \frac{\%CO}{100} \quad \text{etc.}$$

Group I Corrections

The residual gas corrections for the group I experiments are extremely important. A change in gas composition is evident in Table 8.2. The oxygen concentration is decreasing and the carbon monoxide and carbon dioxide concentration are increasing as the test pressures are lowered. At the P_3 gage site which is closest to the specimen the oxygen concentration is reduced to below 20% during the latter part of run # 2. The pressure data given in Table 8.1 for the group 1 runs have been corrected for residual gases. The sensitivity constants for the various gases are as follows:

$$K_{O_2} = P_{obs}/P_{corr}\ ^*$$

$K_{CO} = 1.40$ (From run 11 using ΔW_c)

$K_{CO_2} = 1.45$ (ref (2))

$K_{H_2O} = 1.00$ (From run 10 using ΔWc and ΔWg)

$K_{H_2} = 0.45$ (ref (2))

$K_{He} = 0.17$ (ref (2))

---

*From a regression fit of the calibration data in Appendix 11.1

Group II and Group III Corrections

The analysis of the group II materials (and also the group III materials) is complicated by the presence of large hydrogen accumulations at the P_1 and P_3 gage sites. The attempt to continuously remove hydrogen was only partially successful. A palladum leak and auxiliary pumping system were used at the P_3 site to remove hydrogen as it was formed. Nevertheless, the mass spectrometers indicate that large amounts of hydrogen collected in the system. The data indicates that there was a particularly high build up near the P_1 gage site. This high concentration of hydrogen seems improbable for several reasons.

1. Hydrogen was being pumped away at the P_3 gage location.
2. The path between the specimen and P_1 is longer than between the the specimen and P_3 and is somewhat tortureous.
3. The net gas flow is from P_1 to the specimen.

When the values of gettering rate, flux and sticking probability are corrected for the residuals gases as was done for the Group I materials, the plot of flux verses gettering rate for run # 5 shows marked changes from the expected results. A plot of these data is shown in Figure 9.8. The values of b_1 and b_2 are -1.246 and 0.813 respectively. It would appear that the plot for the uncorrected data as shown in Figure 9.2 more closely follows the established pattern and gives a better fit. In addition, the calculated weight gains for the uncorrected data is closer to the measured weights gain than is the calculated value for the corrected data (see section 9.5).

There are several possible reasons why the uncorrected data provides such a good fit compared to the corrected data. These reasons include:

1. The sensitivity value of 0.45 is wrong for hydrogen. A preliminary investigation of other values shows no other value tried provides a better

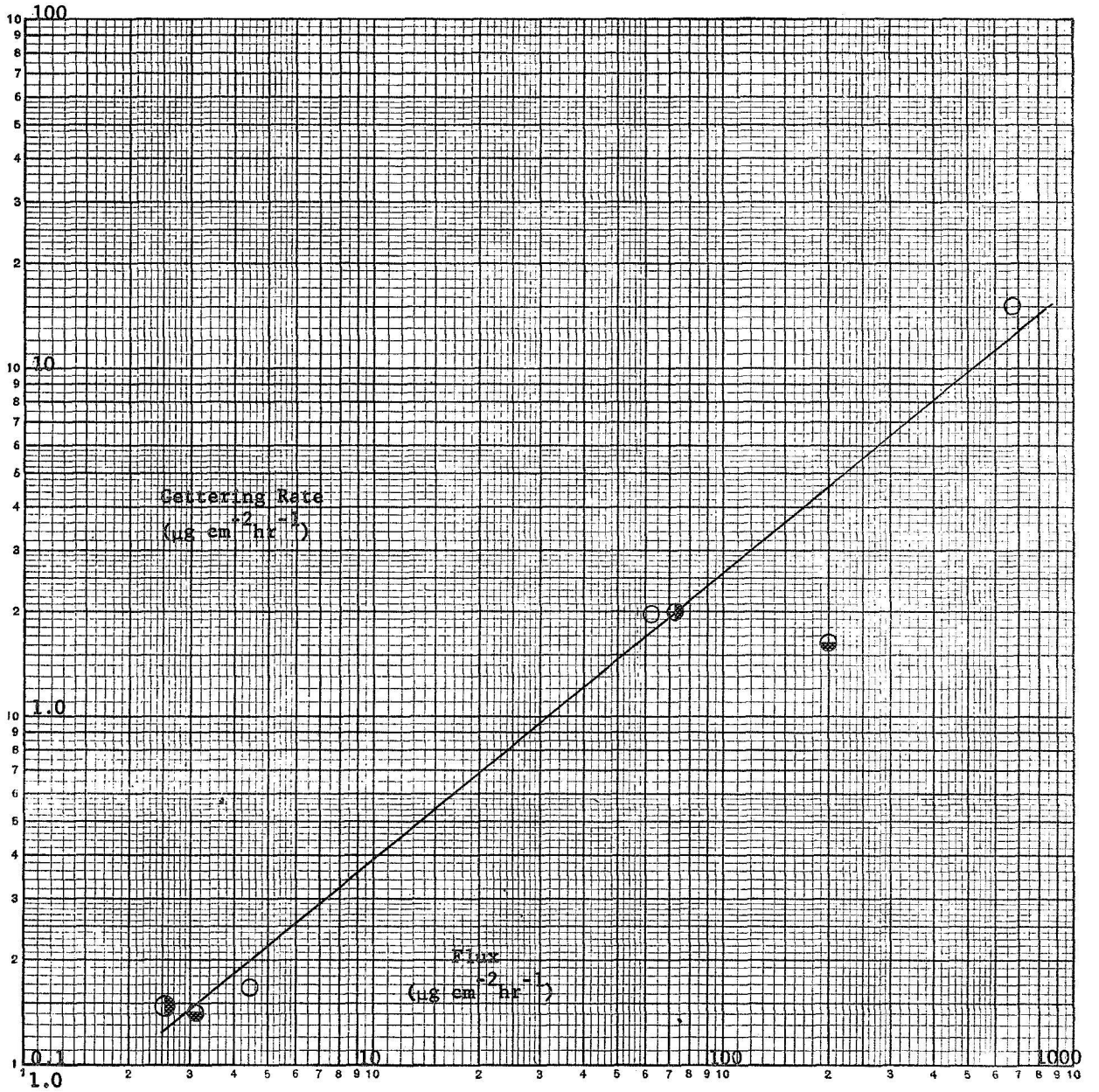


Figure 9.8 Flux Versus Gettering Rate for Water Vapor on Columbium-Zirconium Alloys. Corrected for Residual Gases.

○ 5-Cb-1Zr-2

Open: 1373°K
Right Half Shaded: 1253°K
Lower Half Shaded: 1158°K

fit, although a value of 0.10 appeared promising it still could not approach the fit of the uncorrected data nor match the weight gain estimates.

2. The mass spectrometer tubes do not operate properly in high partial pressures of hydrogen, but the ion vacuum gages are unaffected.
3. Sensitivities of the other gases are changed by large hydrogen partial pressures.

No definite conclusions can be made with the data at hand.

Group IV Corrections

There is no need for residual gas corrections with the Group IV materials since the contaminant, carbon monoxide, accounts for over 90% of the gas content during all phases of the experiment.

The major problem with group IV is determining the correct sensitivity value for carbon monoxide. A value of 1.40 for the sensitivity results in a plot that provides a good fit to the theory except that the points at high flux rates seemed to be in error and the calculated weight gain for run # 12 was much too high. A sensitivity value of 2.00 can be derived from ΔWg instead of ΔWc. This lowers the ΔWp estimate somewhat for run 12 but not significantly. An attempt was made to correct the sensitivity values at the higher pressures and the results are shown in Figure 9.7. The sensitivities were estimated by treating them as points on a log-log plot of P_{true} versus P_{obs} and deriving the line equation

$$\log P_{true} = -.611 + .907 \log P_{obs}$$

Even with these corrections, the weight gain calculation gives only a fair agreement with the measured weight gains for run # 12. In Table 8.1, the values listed are based on a sensitivity of 1.40. It should be pointed out that if

the sensitivity of all three ionization gages are assumed to be the same, regardless of the actual values at various test pressures, then the sticking probabilities will always be the same for a given set of P_0, P_1, and P_3 values for a given specimen. The gettering rates will differ, however.

9.3 Sticking Probability

The sticking probability is the ratio of the number of gas molecules that react with the metal to the total number of gas molecules that impinge upon the metals' surface. The sticking probability is a useful measure of the activity of a particular alloy toward a particular contaminant gas. Oxygen, for example, appears to be more reactive than water vapor when exposed to the hot surface of a columbium - 1% zirconium alloy specimen under low pressure. This is demonstrated by the generally higher sticking probabilities for oxygen when compared to water vapor for the alloy.

The sticking probability is a useful parameter only as long as the surface reaction is not diffusion rate controlled. At low pressures with metal samples of sufficient thickness, the metal will always exhibit a nearly clean surface to the impinging gas. As the pressure is increased and the sample thickness is decreased, a point will be reached where the rate of molecular reaction with the surface is greater than the diffusion rate of the gas into the specimen. At this point, the sticking probability will no longer be applicable to the alloy since it is surface dependent. The initial gas absorption process is a linear function of time. This can demonstrated by plotting the accumulated weight gains during a test run versus time. A plot for several selected runs is shown in Figure 9.9. It is apparent that the plots in Figure 9.9 demonstrate this linear time dependence. All of the experiments run under this contract fulfilled this condition.

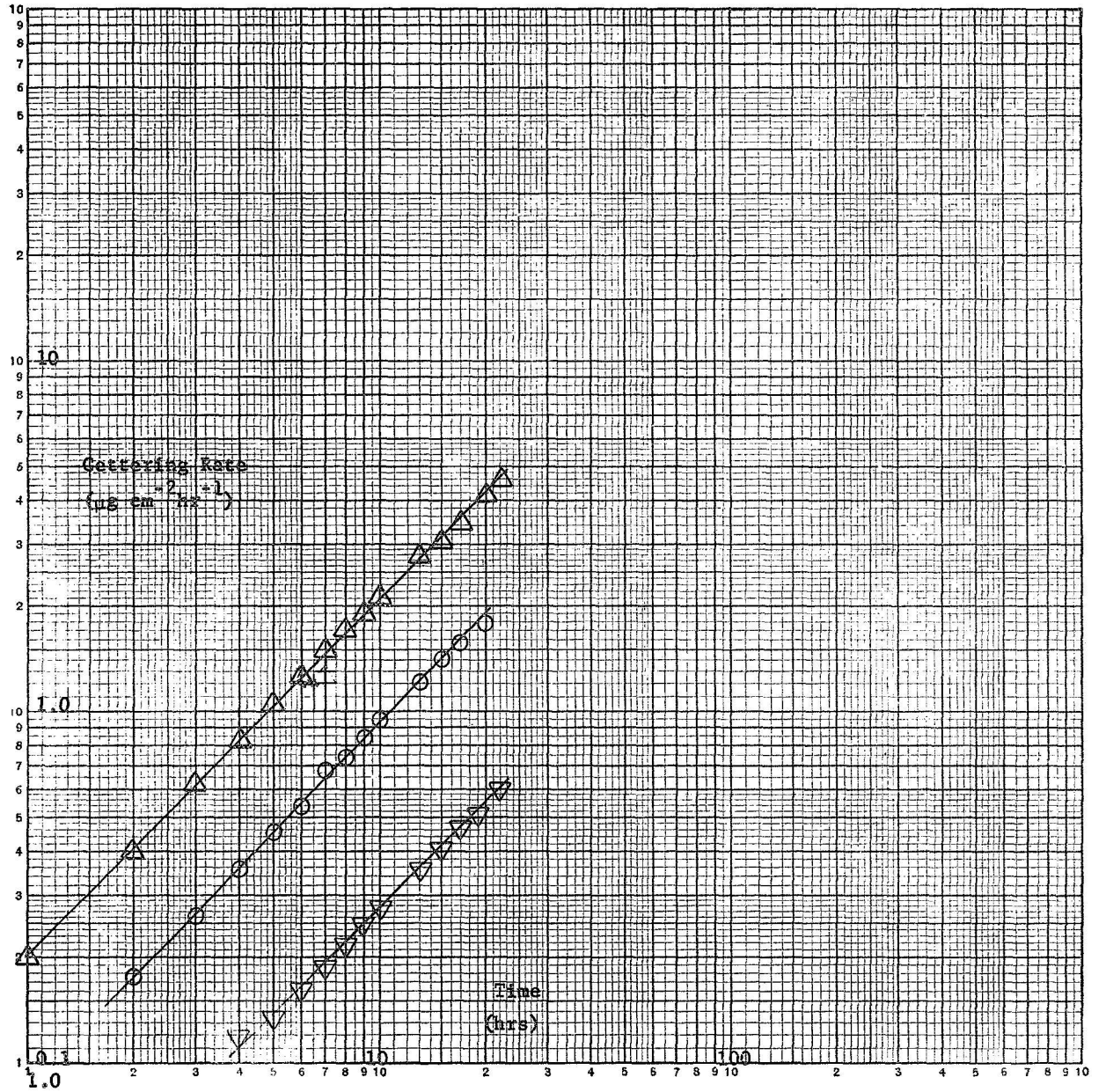


Figure 9.9 Gettering Rate Versus Time for Three Selected Test Run Steps.

△ 2-Cb-1Zr-5-O_2 102 to 214 hours

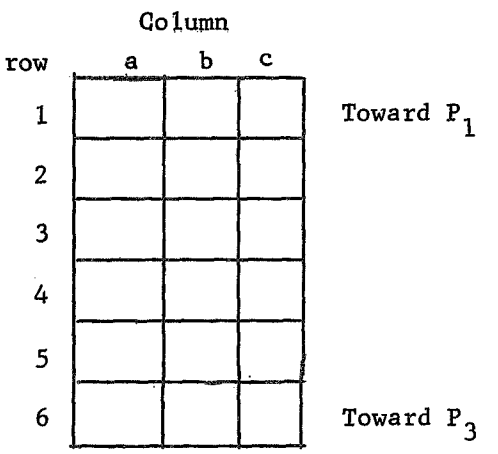
○ 8-Ta-10W-80-H_2O

▽ 12-Ta-78-CO 169 to 192 hours

9.4 Differential Pressure Across the Specimen

The metal specimen used during these experiments pumped the contaminant gas in its immediate surroundings. It is of interest to know if there exists a significant pressure drop across the length of the specimen since the result could influence the corrected pressures and the effective surface area of the specimen.

After the experiments were completed, each specimen was cut into sections. An elemental analysis was made of certain sections that would show a pressure gradient along the length of the specimen. A sectioned sketch of a specimen is shown below locating the positions of the various sections.



The analysis of each specimen is given in Table 9.2. The analysis indicates that there is no significant pressure drop across the specimen, except for run 4 during which the test failed.

Table 9.2 ELEMENTAL ANALYSIS

Sample	Analysis (ppm)				Location
	O_2	N_2	C	H	
1-Cb-1Zr-7	517			< 1	1a
	435			< 1	2a
	421			< 1	3a
	403			< 1	4a
	410			2	5a
	403			1	6a
		12			
			14		3c
			11		4c
2-Cb-1Zr-5	6540	14		< 5	1a
	6100	12		< 5	2a
	5960	11		< 5	3a
	5760	11		< 5	4a
	5720	10		< 5	5a
	5890	10		< 5	6a
			46		1c
			27		2c
			29		4c
3-Cb-.75Zr-1	1.2%			7	1a
	1.23%			6	2a
			35		1b
			52		2b
			28		1c
4-Cb-1Zr-1	2370*	28			1a
	2350	30		< 2	2a
	2030	45		< 2	3a
	1390	59		< 2	4a
	1190	60		3	6a
			24		1b
			17		3b
5-Cb-1Zr-2	1910	9		< 2	1a
	2390	29		< 2	2a
	2160	23		< 2	3a
	2200	20		< 2	4a
	2160	19		2	5a
	2220	19		< 2	6a
			16		2c
			16		4c

*This test was terminated because the ion gage tabulation cracked.

Table 9.2 ELEMENTAL ANALYSIS (con't)

Sample	Analysis (ppm)				Location
	O_2	N_2	C	H	
6-Ta-77	1130	29		< 2	1a
	1110	25		< 2	2a
	1080	25		< 2	3a
	1035	25		< 2	4a
	1050	24		< 2	5a
	1120	23		< 2	6a
			5		1c
			6		2c
7-T111-84	1700	9		< 2	1a
	1530	9		< 2	2a
	1600	10		< 2	3a
	1610	7		< 2	4a
	1600	7		< 2	5a
	1670	5		< 2	6a
			12		2c
			8		4c
8-Ta-10W-80	996	18		< 2	2a
	1026	13		< 2	3a
	1040	12		< 2	4a
	984	13		< 2	5a
	1068	12		< 2	6a
			12		2c
			5		3c
			23		4c
9-Ta-2Hf	1400	21		< 2	1a
	1430	16		< 2	2a
	1400	20		< 2	3a
	1390	15		< 2	4a
	1420	11		< 2	5a
	1410	11		< 2	6a
			5		3c
			8		4c
10-T-111-85	3080	11		3	1a
	3110	8		< 2	2a
	3100	9		2	3a
	3100	11		< 2	4a
	3150	12		2	5a
	3190	12		2	6a
			2		2c
			21		4c

Table 9.2 ELEMENTAL ANALYSIS (con't)

Sample	Analysis (ppm)				Location
	O_2	N_2	C	H	
11-Ta-76	173	26		< 2	1a
	181	26		< 1	2a
	183	23		< 1	3a
	177	23		< 1	4a
	167	21		1	5a
	192	23		1	6a
			152		1b
			108		2b
			119		3b
			113		4b
			113		5b
			113		6b
12-Ta-78	192	20		1	1a
	192	25		1	2a
	166	26		< 1	3a
	175	17		1	4a
	193	23		1	5a
	180	22		< 1	6a
			119		1b
			129		2b
			123		3b
			122		4b
			127		5b
			111		6b

9.5 Experimental Verification of Gas Absorption Rate

The pressure drop method of establishing gettering rates can be used to measure very small chemisorbed gas weight increases in the specimen. The total weight gain of a sample during a test run can be computed by summing all of the accumulated weight gains and multiplying by the sample's surface area. The result of this computation can then be compared with weight gains measured by other methods. Table 9.3 gives the total weight gains of all samples determined by the pressure drop method (ΔW_p) and compares them with the weight gains as found by two other methods. These two methods are gravimetric analysis (ΔW_g) and gas analysis (ΔW_c). Generally, the agreement between the three methods is quite good.

Table 9.3 Total Weight Gains

Samples	ΔWg(mg)	ΔWc(mg)	ΔWp(mg)
1-Cb-1Zr-7	2.51	2.74	3.00[1]
2-Cb-1Zr-5	56.33	56.34	54.81[1]
3-Cb-.75Zr-1	42.90	39.26	54.72[1]
4-Cb-1Zr-1	15.87	15.33	-
5-Cb-1Zr-2	20.41	19.36	21.20
6-Ta-77	14.09	14.38	16.92
7-T-111-84	16.87	21.69	18.80
8-Ta-10W-80	13.90	13.66	17.67
9-Ta-2Hf-1	17.30	17.75	19.68
10-T-111-85	43.09	42.26	43.32
11-Ta-76	2.62	3.74	3.70[3] or 2.59[4]
12-Ta-78	3.07	3.72	10.43[2]
			14.15[3]
			9.91[4]

[1] Corrected for residual gases, K_{O_2} (ion gages) from calibration (appendix 11.1)

[2] Variable sensitivity see section 9.2 Group IV, $K_{H_2O} = 1.00$

[3] $K_{CO} = 1.4$

[4] $K_{CO} = 2.0$

10.0 CONCLUSIONS

The investigation of absorption rate sticking probabilities for oxygen, water vapor and carbon monoxide on tantalum and columbium alloys yields the following conclusions.

1. The sticking probability between 1158 and 1373°K for oxygen on nominal columbium 1% zirconium is 0.129 and agrees well with the available literature. Gage corrections for residual gases were necessary.
2. The sticking probability between 1158 and 1373°K for water vapor on nominal columbium 1% zirconium is 5.907×10^{-3}. No corrections for residual gases were made even though there was a significant build up of hydrogen in the system.
3. The sticking probability for water vapor on various tantalum alloys between 1158 and 1373°K are as follows:

Tantalum	4.057×10^{-3}
Ta-8%W-2%Hf	4.695×10^{-3}
Ta-10%W	3.644×10^{-3}
Ta-2%Hf	6.450×10^{-3}

   No gage correction for residual gases were necessary
4. The sticking probability between 1158 and 1373°K for carbon monoxide on tantalum is 2.328×10^{-3}. No gage corrections for residual gases were necessary. A gage sensitivity for carbon monoxide of 1.40 was used.
5. The sticking probability has little or no dependence upon specimen temperature over the range from 1158 to 1373°K.

6. The total weight gains of the samples as calculated by the pressure drop method are in good agreement with the weight gains measured by other methods.

11.0 APPENDIX

11.1 Ion Gage Calibrations For Oxygen

The three ion gages used in each system (referred to as the "system" ion gages) were calibrated in the following manner. First, two ion gage tubes and their respective control panels (referred to as the "secondary standard ion gages) were sent to an independent laboratory for calibration. Each "secondary standard" ion gage tube was accompanied by its own individual control panel so that the characteristics of the control panel - as well as the gage tube - would be included in the calibration. The calibration of two secondary standards permitted periodic checks of one against the other, increasing the confidence level of each subsequent gage calibration; in addition, the risk of losing the calibration because of the malfunction of a single tube or panel was reduced.

After calibration of the secondary standards, both secondary standards and the three system gages were mounted in close proximity on a manifold as shown in Figure 11.1. The reason for close-mounting the gages is to reduce error from either gage pumping or system outgassing. Readings from all five gages were then taken at several steady-state pressure levels in each decade of pressure. The "true" pressure at each level was then taken as the arithmetic average of the corrected pressures of the two secondary standards.

There were three different calibrations used during the program. Each is described in the Sections which follow.

Calibration of System Gages

During the program, four sets of three ion gages were calibrated. These sets will be distinguished by the labels first set, second set, third set, and fourth set. The data are given in Tables 11.1 through 11.7 and the calibration curves in Figures 11.2 through 11.13.

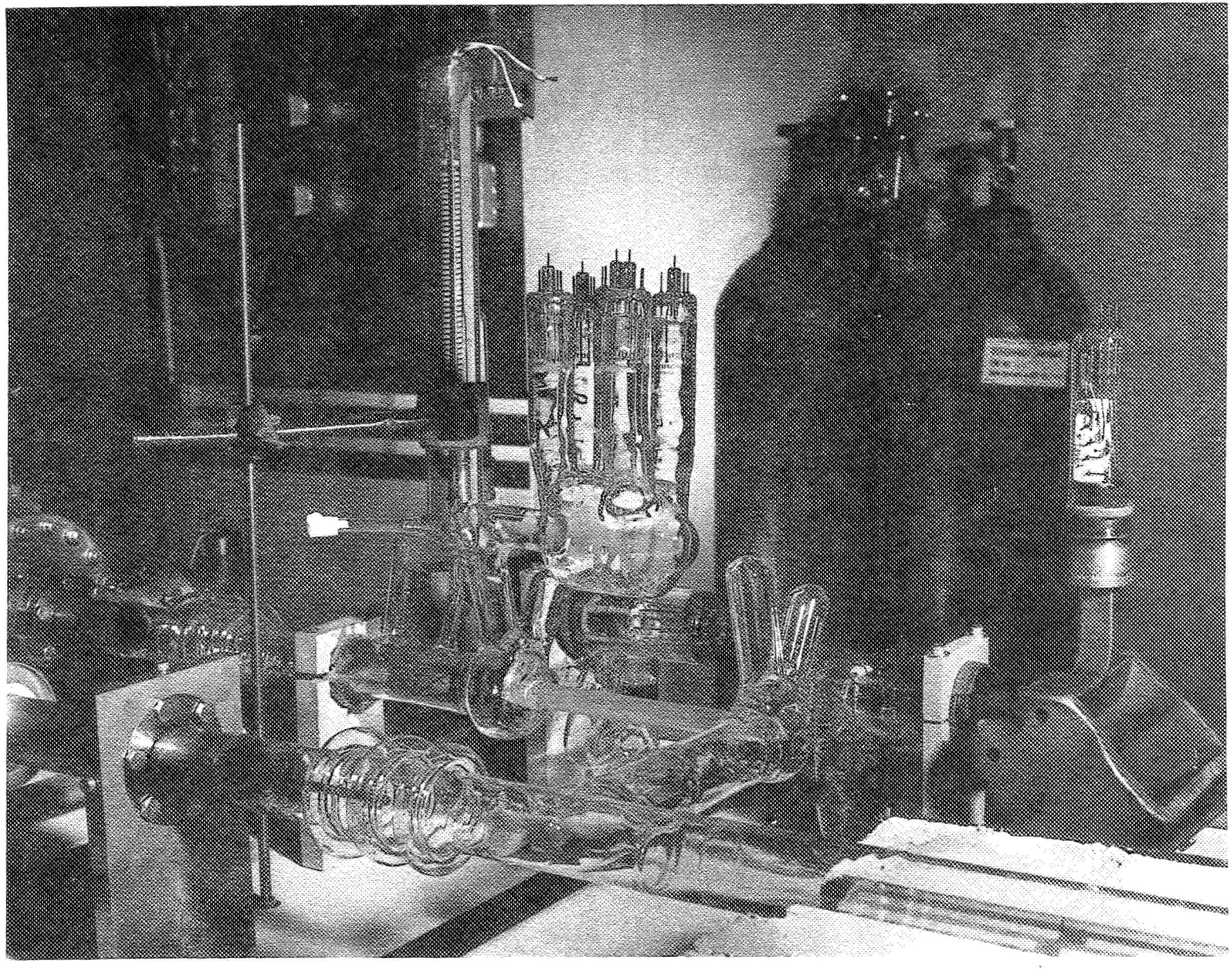


Figure 11. 1. Ion Gage Calibration Cluster

TABLE 11.1

First Set of Ionization Gages

CALIBRATION OF 20 OCTOBER 1966

#10 Indicated	#10 True AVCO CAL.	#11 Indicated	#11 True AVCO CAL.	Avg. True	Quartz Indicated	Pump Indicated	Sample Indicated
7.10 (-5)	*	6.40 (-5)	*	*	1.10 (-4)	9.30 (-5)	1.10 (-4)
4.90 (-5)	7.50 (-5)	4.50 (-5)	8.20 (-5)	7.85 (-5)	8.00 (-5)	6.50 (-5)	7.70 (-5)
3.70 (-5)	5.80 (-5)	3.60 (-5)	6.60 (-5)	6.20 (-5)	6.10 (-5)	5.00 (-5)	6.05 (-5)
2.40 (-5)	3.90 (-5)	2.30 (-5)	4.20 (-5)	4.05 (-5)	3.95 (-5)	3.20 (-5)	3.90 (-5)
1.55 (-5)	2.55 (-5)	1.40 (-5)	2.60 (-5)	2.57 (-5)	2.50 (-5)	2.00 (-5)	2.40 (-5)
8.20 (-6)	1.20 (-5)	7.60 (-6)	1.30 (-5)	1.25 (-5)	1.30 (-5)	1.10 (-5)	1.30 (-5)
5.40 (-6)	8.10 (-6)	5.30 (-6)	9.10 (-6)	8.60 (-6)	9.00 (-6)	7.50 (-6)	9.05 (-6)
4.20 (-6)	6.40 (-6)	4.10 (-6)	7.10 (-6)	6.75 (-6)	6.90 (-6)	5.70 (-6)	7.00 (-6)
3.30 (-6)	5.10 (-6)	3.20 (-6)	5.60 (-6)	5.35 (-6)	5.30 (-6)	4.30 (-6)	5.35 (-6)
2.10 (-6)	3.30 (-6)	2.00 (-6)	3.50 (-6)	3.40 (-6)	3.40 (-6)	2.80 (-6)	3.45 (-6)
1.10 (-6)	1.80 (-6)	9.50 (-7)	1.65 (-6)	1.73 (-6)	1.70 (-6)	1.30 (-6)	1.70 (-6)
5.50 (-7)	8.10 (-7)	5.40 (-7)	9.60 (-7)	8.85 (-7)	9.19 (-7)	7.60 (-7)	9.30 (-7)
4.30 (-7)	6.50 (-7)	4.20 (-7)	7.50 (-7)	7.00 (-7)	7.10 (-7)	6.00 (-7)	7.30 (-7)
2.80 (-7)	4.40 (-7)	2.80 (-7)	5.10 (-7)	4.75 (-7)	4.80 (-7)	3.95 (-7)	4.90 (-7)
1.60 (-7)	2.60 (-7)	1.60 (-7)	3.00 (-7)	2.80 (-7)	2.80 (-7)	2.30 (-7)	2.90 (-7)
9.60 (-8)	*	8.50 (-8)	*	*	1.60 (-7)	1.30 (-7)	1.70 (-7)
4.80 (-8)	*	4.30 (-8)	*	*	8.40 (-8)	6.40 (-8)	8.00 (-8)
3.10 (-8)	*	2.80 (-8)	*	*	5.60 (-8)	4.30 (-8)	5.40 (-8)
2.60 (-8)	*	2.40 (-8)	*	*	4.80 (-8)	3.60 (-8)	4.60 (-8)

Note: Pressures are in torr. Numbers in parentheses are powers of 10.

* Calibration not made.

TABLE 11.2

First Set of Ionization Gages

CALIBRATION OF 24 OCTOBER 1966

#10 Indicated	#10 True AVCO Cal.	#11 Indicated	#11 True AVCO Cal.	Avg. True	Quartz Indicated	Pump Indicated	Sample Indicated
6.20 (-5)	9.30 (-5)	5.70 (-5)	10.05 (-5)	9.68 (-5)	9.80 (-5)	8.20 (-5)	9.90 (-5)
5.30 (-5)	8.10 (-5)	4.95 (-5)	9.00 (-5)	8.55 (-5)	8.60 (-5)	7.10 (-5)	8.60 (-5)
3.80 (-5)	5.90 (-5)	3.60 (-5)	6.60 (-5)	6.25 (-5)	6.30 (-5)	5.10 (-5)	6.20 (-5)
2.70 (-5)	4.30 (-5)	2.60 (-5)	4.80 (-5)	4.55 (-5)	4.40 (-5)	3.60 (-5)	4.40 (-5)
1.50 (-5)	2 50 (-5)	1.40 (-5)	2.60 (-5)	2.55 (-5)	2.40 (-5)	2.00 (-5)	2.40 (-5)
6.00 (-6)	8.90 (-6)	5.60 (-6)	9.60 (-6)	9.25 (-6)	9.60 (-6)	8.00 (-6)	9.80 (-6)
5.30 (-6)	8.00 (-6)	5.10 (-6)	8.80 (-6)	8.40 (-6)	8.70 (-6)	7.20 (-6)	8.80 (-6)
3.40 (-6)	5.30 (-6)	3.30 (-6)	5.70 (-6)	5.50 (-6)	5.60 (-6)	4.50 (-6)	5.60 (-6)
1.80 (-6)	2.95 (-6)	1.70 (-6)	3.00 (-6)	2.97 (-6)	2.80 (-6)	2.30 (-6)	2.90 (-6)
1.10 (-6)	1.85 (-6)	9.40 (-7)	1.65 (-6)	1.75 (-6)	1.60 (-6)	1.40 (-6)	1.60 (-6)
6.20 (-7)	7.70 (-7)	5.10 (-7)	9.00 (-7)	8.35 (-7)	8.70 (-7)	7.20 (-7)	8.90 (-7)
3.30 (-7)	5.10 (-7)	3.20 (-7)	5.80 (-7)	6.45 (-7)	5.40 (-7)	4.50 (-7)	5.60 (-7)
2.70 (-7)	4.30 (-7)	2.60 (-7)	4.80 (-7)	4.55 (-7)	4.50 (-7)	3.70 (-7)	4.70 (-7)
2.00 (-7)	3.20 (-7)	2.00 (-7)	3.70 (-7)	3.45 (-7)	3.40 (-7)	2.80 (-7)	3.50 (-7)
1.20 (-7)	2.00 (-7)	1.15 (-7)	2.20 (-7)	2.10 (-7)	2.00 (-7)	1.60 (-7)	2.00 (-7)

Note: Pressures are in torr. Numbers in parentheses are powers of 10.

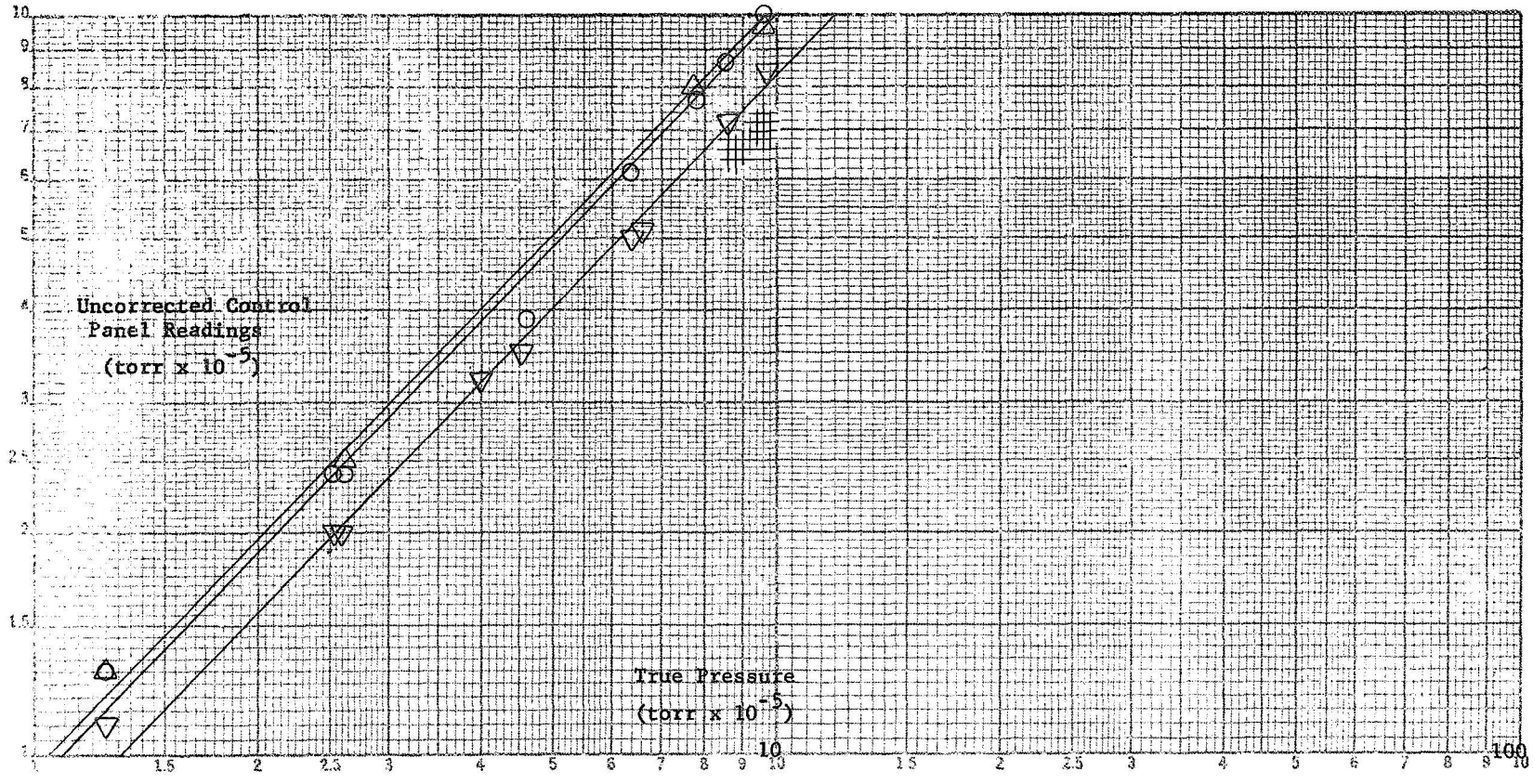


Figure 11.2 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. First Set of Gages, 10^{-5} torr range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel # 50004

○ Sample Ion Gage (P_1) with G.E. Control Panel # 40012

△ Quartz Ion Gage (P_3) with G.E. Control Panel # 40011

Emission Current: 1.0 mA
Calibration Date: 20 October and 24 October, 1966

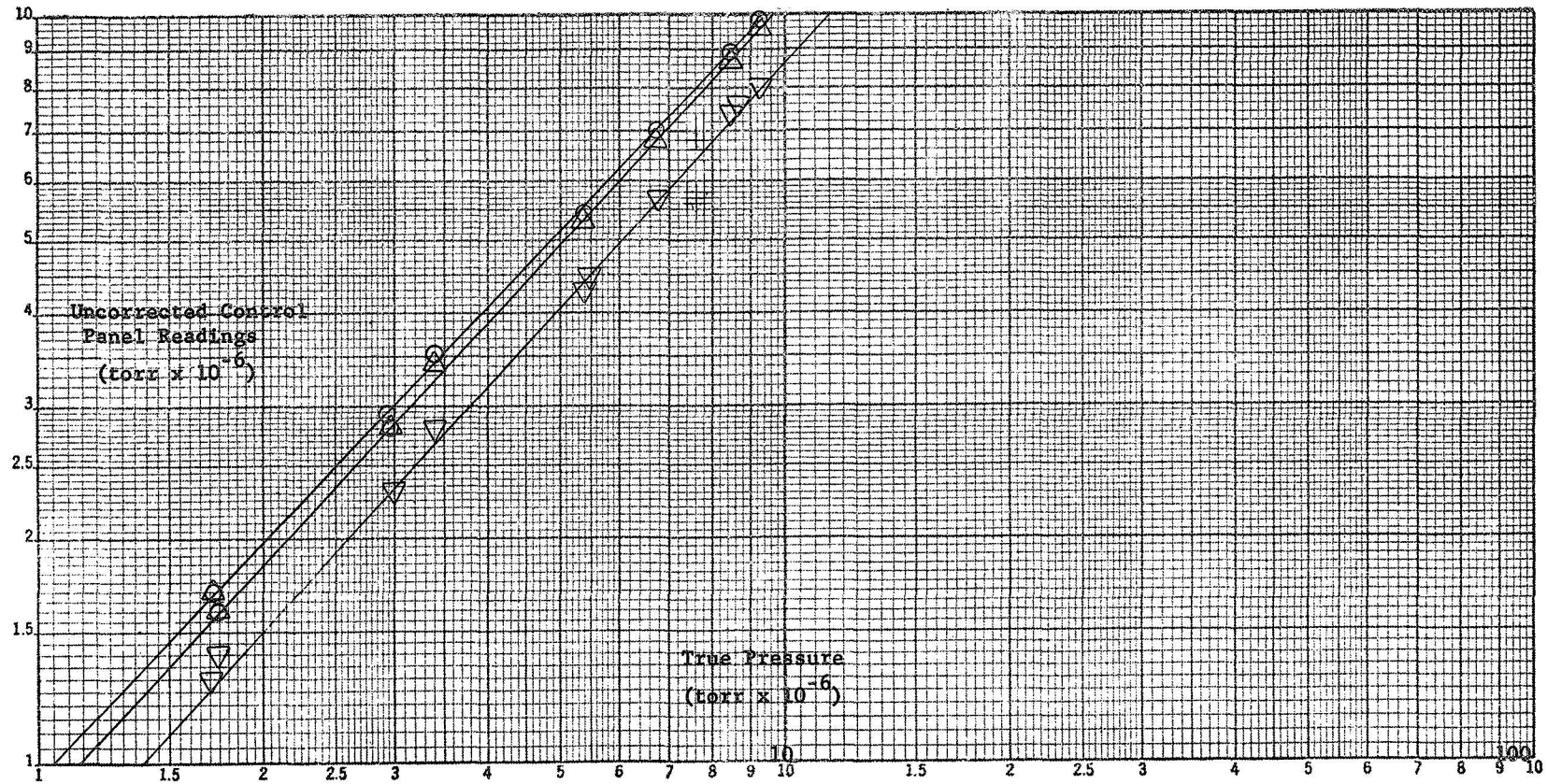


Figure 11.3 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. First Set of Gages, 10^{-6} torr range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel #50004

○ Sample Ion Gage (P_1) with G.E. Control Panel #40012

△ Quartz Ion Gage (P_3) with G.E. Control Panel #40011

Emission Current: 1.0 mA
Calibration Date: 20 October and 24 October, 1966

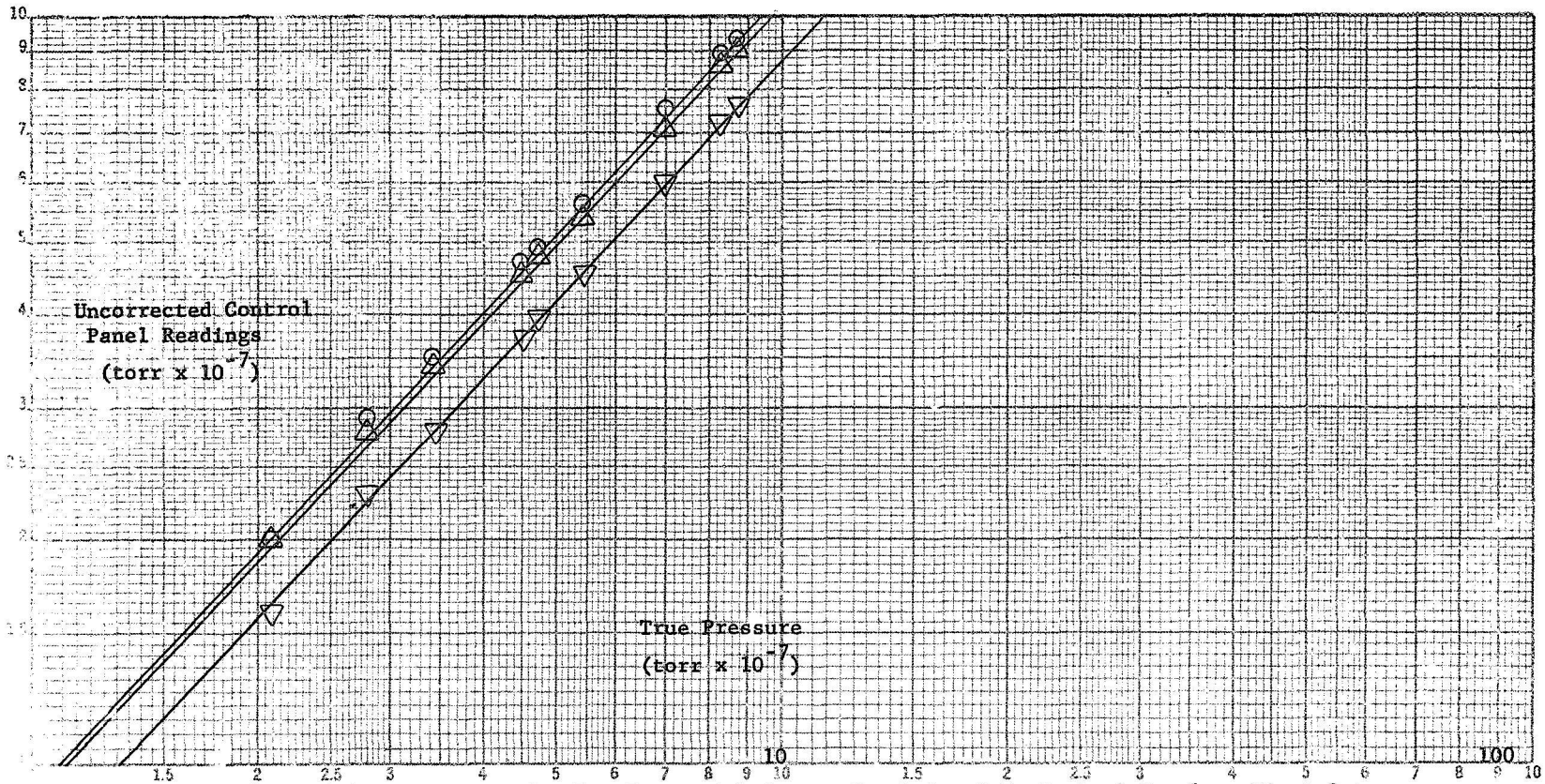


Figure 11.4 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. First Set of Gages, 10^{-7} torr range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel #50004

○ Sample Ion Gage (P_1) with G.E. Control Panel #40012

△ Quartz Ion Gage (P_3) with G.E. Control Panel #40011

Emission Current: 1.0 mA
Calibration Date: 20 October and 24 October, 1966.

TABLE 11.3

Second Set of Ionization Gages

CALIBRATION OF 25 NOVEMBER 1966

#10 Indicated	#10 True AVCO Cal.	#11 Indicated	#11 True AVCO Cal.	Avg. True	Quartz Indicated	Pump Indicated	Sample Indicated
7.6 (-5)	1.13 (-4)	7.0 (-5)	1.27 (-4)	1.20 (-4)	8.0 (-5)	8.2 (-5)	1.0 (-4)
6.7 (-5)	10.00 (-5)	6.1 (-5)	1.10 (-4)	1.05 (-4)	6.9 (-5)	7.0 (-5)	8.8 (-5)
5.0 (-5)	7.70 (-5)	4.6 (-5)	8.40 (-5)	8.05 (-5)	5.2 (-5)	5.2 (-5)	6.7 (-5)
3.5 (-5)	5.50 (-5)	3.3 (-5)	6.10 (-5)	5.80 (-5)	3.7 (-5)	3.7 (-5)	4.8 (-5)
2.0 (-5)	3.25 (-5)	1.9 (-5)	3.50 (-5)	3.38 (-5)	2.1 (-5)	2.1 (-5)	2.6 (-5)
9.2 (-6)	1.33 (-5)	8.2 (-6)	1.40 (-5)	1.36 (-5)	9.4 (-6)	9.6 (-6)	1.2 (-5)
7.1 (-6)	1.05 (-5)	6.4 (-6)	1.10 (-5)	1.07 (-5)	7.2 (-6)	7.2 (-6)	9.2 (-6)
5.4 (-6)	8.10 (-6)	5.0 (-6)	8.60 (-6)	8.35 (-6)	5.6 (-6)	5.6 (-6)	7.1 (-6)
3.4 (-6)	5.30 (-6)	3.2 (-6)	5.60 (-6)	5.45 (-6)	3.5 (-6)	3.5 (-6)	4.5 (-6)
2.0 (-6)	3.20 (-6)	1.9 (-6)	3.30 (-6)	3.25 (-6)	2.0 (-6)	2.0 (-6)	2.6 (-6)
8.6 (-7)	1.24 (-6)	7.9 (-7)	1.38 (-6)	1.33 (-6)	8.8 (-7)	8.9 (-7)	1.2 (-6)
7.1 (-7)	1.03 (-6)	6.6 (-7)	1.15 (-6)	1.09 (-6)	7.3 (-7)	7.4 (-7)	9.4 (-7)
5.8 (-7)	8.70 (-7)	5.5 (-7)	9.70 (-7)	9.30 (-7)	5.9 (-7)	6.0 (-7)	7.6 (-7)
3.6 (-7)	5.50 (-7)	3.5 (-7)	6.30 (-7)	5.90 (-7)	3.8 (-7)	3.8 (-7)	4.9 (-7)
2.8 (-7)	4.40 (-7)	2.7 (-7)	4.95 (-7)	4.70 (-7)	2.9 (-7)	3.0 (-7)	3.8 (-7)

Note: Pressures are in torr. Numbers in parentheses are powers of 10.

TABLE 11.4

Second Set of Ionization Gages

CALIBRATION OF 28 NOVEMBER 1966

#10 Indicated	# 10 True AVCO Cal.	E11 Indicated	#11 True AVCO Cal.	Avg. True	Quartz Indicated	Pump Indicated	Sample Indicated
7.8 (-5)	1.17 (-4)	7.1 (-5)	1.30 (-4)	1.24 (-4)	8.0 (-5)	8.05 (-5)	1.0 (-4)
5.9 (-5)	8.90 (-5)	5.5 (-5)	10.00 (-5)	9.45 (-5)	6.2 (-5)	6.20 (-5)	7.9 (-5)
4.6 (-5)	7.10 (-5)	4.2 (-5)	7.70 (-5)	7.40 (-5)	4.8 (-5)	4.80 (-5)	6.1 (-5)
3.1 (-5)	4.90 (-5)	2.9 (-5).	5.30 (-5)	5.10 (-5)	3.3 (-5)	3.30 (-5)	4.1 (-5)
1.7 (-5)	2.80 (-5)	1.6 (-5)	2.95 (-5)	2.88 (-5)	1.8 (-5)	1.80 (-5)	2.3 (-5)
9.1 (-6)	1.32 (-5)	8.2 (-6)	1.40 (-5)	1.36 (-5)	9.3 (-6)	9.30 (-6)	1.1 (-5)
6.5 (-6)	9.60 (-6)	6.0 (-6)	1.04 (-5)	10.00 (-6)	6.6 (-6)	6.70 (-6)	8.5 (-6)
3.9 (-6)	6.00 (-6)	3.7 (-6)	6.40 (-6)	6.20 (-6)	4.0 (-6)	4.00 (-6)	5.1 (-6)
2.8 (-6)	4.40 (-6)	2.7 (-6)	4.70 (-6)	4.55 (-6)	2.9 (-6)	3.00 (-6)	3.7 (-6)
1.8 (-6)	2.90 (-6)	1.7 (-6)	3.00 (-6)	2.95 (-6)	1.8 (-6)	1.90 (-6)	2.3 (-6)
9.0 (-7)	1.30 (-6)	8.3 (-7)	1.45 (-6)	1.38 (-6)	9.0 (-7)	9.40 (-7)	1.1 (-6)
7.8 (-7)	1.13 (-6)	7.2 (-7)	1.25 (-6)	1.21 (-6)	7.9 (-7)	8.00 (-7)	10.0 (-7)
5.7 (-7)	8.40 (-7)	5.4 (-7)	9.50 (-7)	8.95 (-7)	5.8 (-7)	5.90 (-7)	7.6 (-7)
4.0 (-7)	6.10 (-7)	3.8 (-7)	6.90 (-7)	6.50 (-7)	4.1 (-7)	4.20 (-7)	5.4 (-7)
3.1 (-7)	4.80 (-7)	3.1 (-7)	5.60 (-7)	5.20 (-7)	3.2 (-7)	3.40 (-7)	4.3 (-7)

Note: Pressures are in torr. Numbers in parentheses are powers of 10.

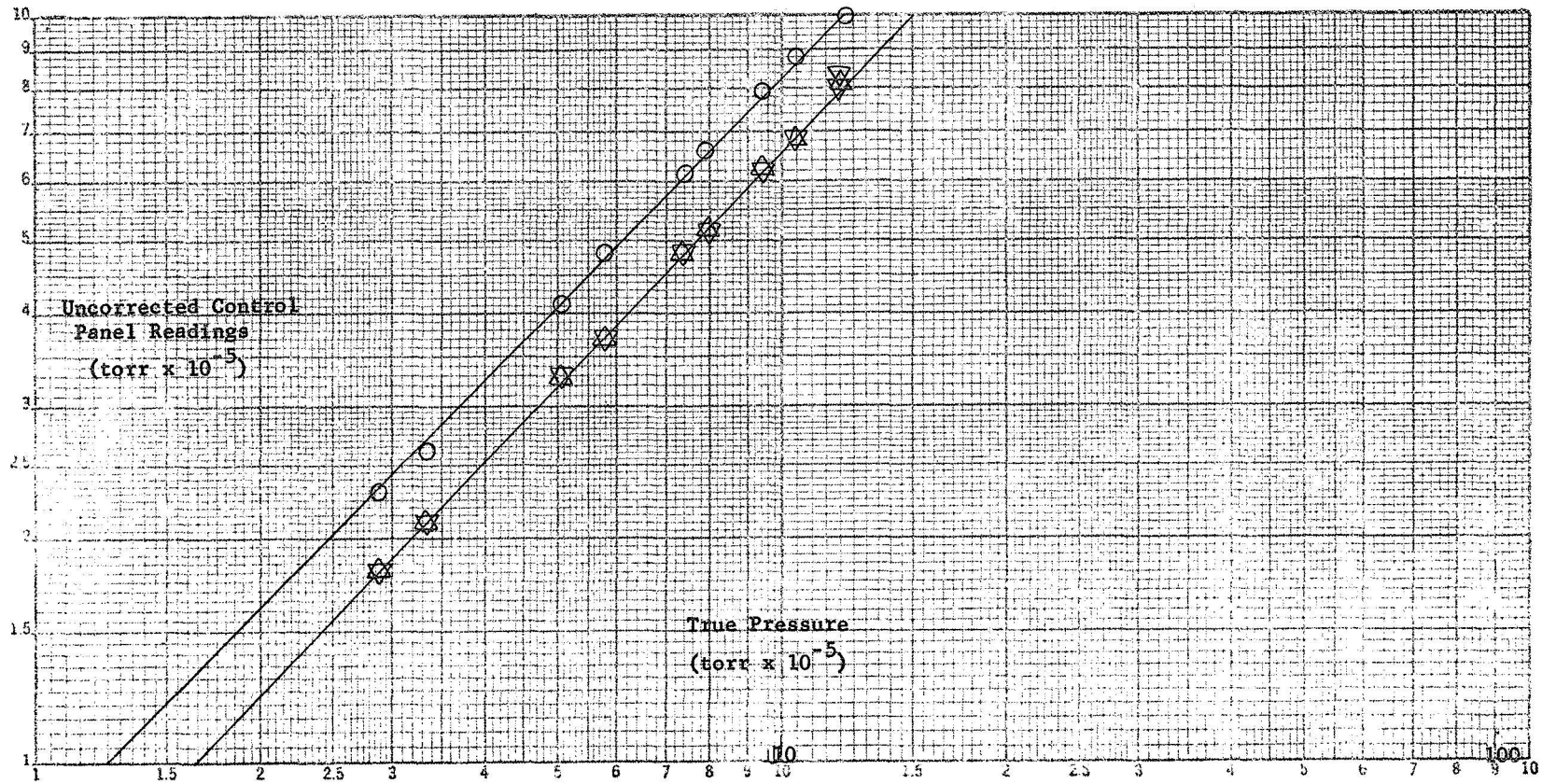


Figure 11.5 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. Second Set of Gages, 10^{-5} torr range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel #50004

○ Sample Ion Gage (P_1) with G.E. Control Panel #40012 Emission Current: 1.0 mA

△ Quartz Ion Gage (P_3) with G.E. Control Panel #40011 Calibration Date: 25 November and 28 November, 1966

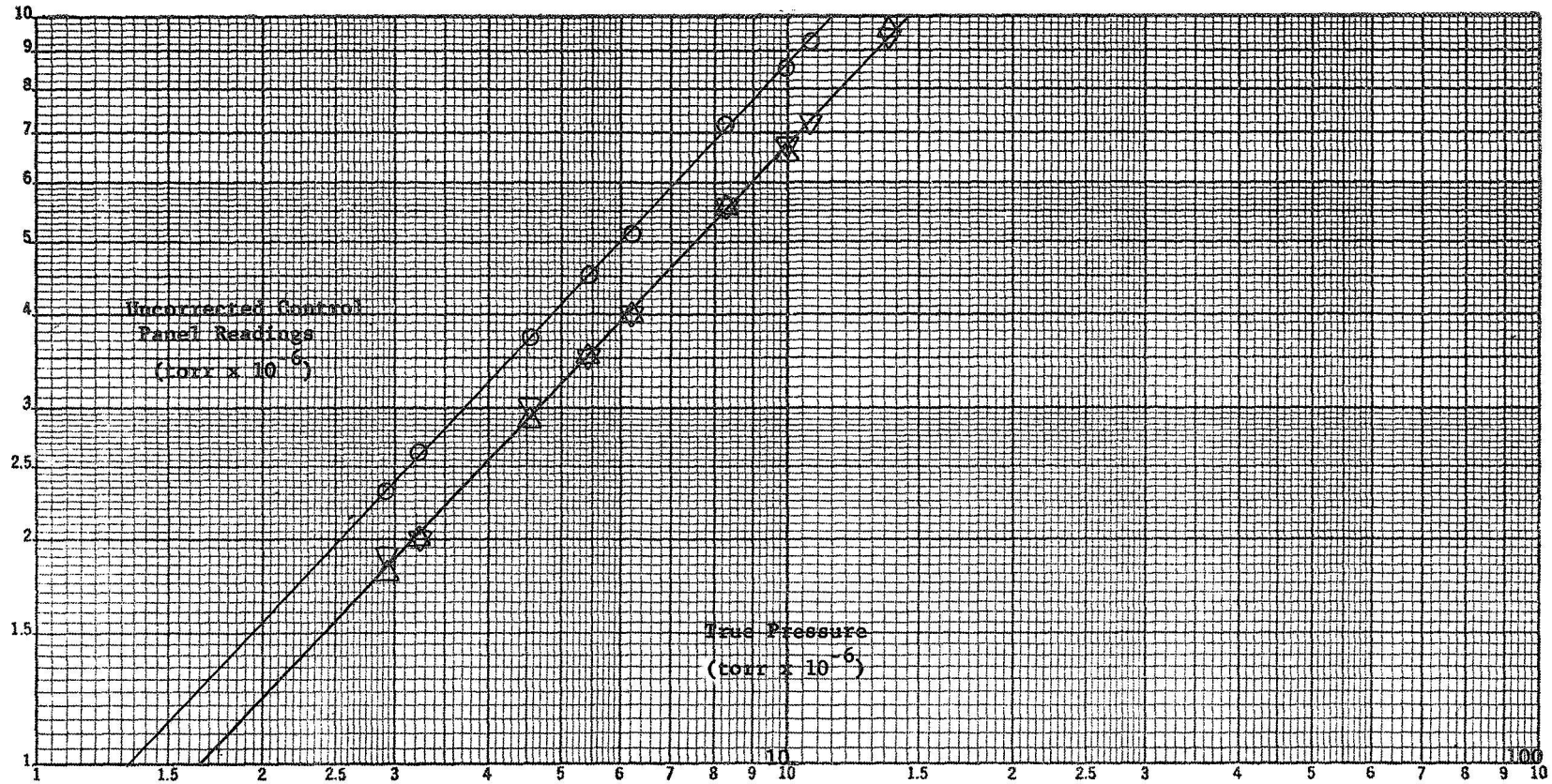


Figure 11.6 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. Second Set of Gases, 10^{-6} torr range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel #50004

○ Sample Ion Gage (P_1) with G.E. Control Panel #40012 Emission Current: 1.0 mA

△ Quartz Ion Gage (P_3) with G.E. Control Panel #40011 Calibration Date: 25 November and 28 November, 1966

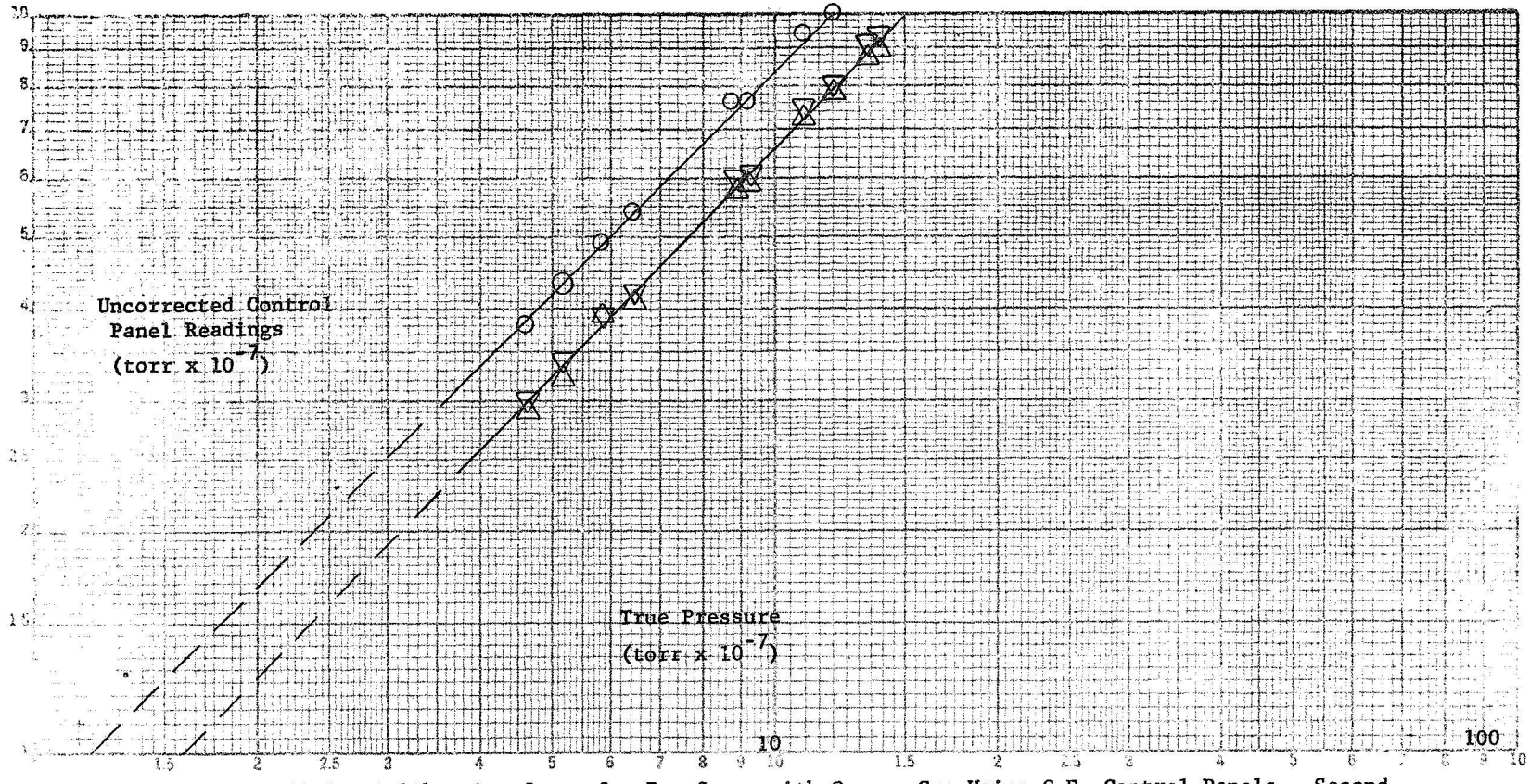


Figure 11.7 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. Second Set of Gages, 10^{-7} torr range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel #50004

○ Sample Ion Gage (P_1) with G.E. Control Panel #40012 Emission Current: 1.0 mA

△ Quartz Ion Gage (P_3) with G.E. Control Panel #40011 Calibration Date: 25 November and 28 November, 1966

TABLE 11.5

Third Set of Ionization Gages

CALIBRATION - March 22, 1967

#10 Indicated	#10 True AVCO Cal.	#11 Indicated	#11 True AVCO Cal.	Average True	Quartz Indicated	Pump Indicated	Sample Indicated
9.6 (-5)	1.42 (-4)	8.5 (-5)	1.55 (-4)	1.49 (-4)	9.3 (-5)	1.1 (-4)	8.9 (-5)
7.2 (-5)	1.08 (-4)	6.4 (-5)	1.17 (-4)	1.12 (-4)	6.9 (-5)	8.2 (-5)	6.5 (-5)
5.7 (-5)	8.70 (-5)	5.2 (-5)	9.50 (-5)	9.10 (-5)	5.4 (-5)	6.4 (-5)	5.0 (-5)
4.6 (-5)	7.10 (-5)	4.2 (-5)	7.70 (-5)	7.40 (-5)	4.5 (-5)	5.3 (-5)	4.2 (-5)
3.3 (-5)	5.20 (-5)	3.1 (-5)	5.70 (-5)	5.50 (-5)	3.2 (-5)	3.7 (-5)	2.9 (-5)
2.0 (-5)	3.25 (-5)	1.8 (-5)	3.30 (-5)	3.28 (-5)	1.9 (-5)	2.3 (-5)	1.8 (-5)
10.0 (-6)	1.45 (-5)	8.7 (-6)	1.50 (-5)	1.48 (-5)	9.0 (-6)	1.1 (-5)	8.8 (-6)
8.0 (-6)	1.17 (-5)	7.0 (-6)	1.20 (-5)	1.19 (-5)	7.2 (-6)	9.0 (-6)	7.0 (-6)
5.5 (-6)	8.20 (-6)	5.0 (-6)	8.60 (-6)	8.40 (-6)	5.0 (-6)	6.3 (-6)	4.9 (-6)
3.8 (-6)	5.85 (-6)	3.6 (-6)	6.25 (-6)	6.05 (-6)	3.6 (-6)	4.6 (-6)	3.5 (-6)
2.8 (-6)	4.40 (-6)	2.6 (-6)	4.55 (-6)	4.48 (-6)	2.6 (-6)	3.2 (-6)	2.5 (-6)
2.1 (-6)	3.40 (-6)	1.9 (-6)	3.30 (-6)	3.35 (-6)	1.9 (-6)	2.4 (-6)	1.9 (-6)
9.1 (-7)	1.30 (-6)	8.1 (-7)	1.40 (-6)	1.35 (-6)	8.1 (-7)	1.1 (-6)	8.4 (-7)
6.3 (-7)	9.20 (-7)	5.8 (-7)	1.03 (-6)	9.80 (-7)	5.7 (-7)	7.4 (-7)	5.9 (-7)
5.6 (-7)	8.30 (-7)	5.2 (-7)	9.20 (-7)	8.80 (-7)	5.1 (-7)	6.5 (-7)	5.3 (-7)
4.8 (-7)	7.20 (-7)	4.5 (-7)	8.00 (-7)	7.60 (-7)	4.4 (-7)	5.6 (-7)	4.6 (-7)
3.4 (-7)	5.20 (-7)	3.3 (-7)	6.00 (-7)	5.60 (-7)	3.2 (-7)	4.0 (-7)	3.3 (-7)
2.5 (-7)	3.90 (-7)	2.4 (-7)	4.40 (-7)	4.20 (-7)	2.3 (-7)	3.0 (-7)	2.4 (-7)

Note: Pressures are in torr. Numbers in parentheses are powers of 10.

TABLE 11.6

Third Set of Ionization Gages

CALIBRATION - MARCH 23, 1967

#10 Indicated	#10 True AVCO Cal.	#11 Indicated	#11 True AVCO Cal.	Avg. True	Quartz Indicated	Pump Indicated	Sample Indicated
9.5 (-5)	1.40 (-4)	8.5 (-5)	1.55 (-4)	1.48 (-4)	9.3 (-5	1.1 (-4)	8.9 (-5)
8.6 (-5)	1.28 (-4)	7.6 (-5)	1.40 (-4)	1.34 (-4)	8.5 (-5)	9.9 (-5)	8.1 (-5)
5.2 (-5)	7.90 (-5)	4.7 (-5)	8.50 (-5)	8.20 (-5)	5.1 (-5)	6.0 (-5)	4.8 (-5)
3.9 (-5)	6.10 (-5)	3.6 (-5)	6.60 (-5)	6.40 (-5)	3.8 (-5)	4.6 (-5)	3.6 (-5)
2.4 (-5)	3.90 (-5)	2.3 (-5)	4.20 (-5)	4.05 (-5)	2.4 (-5)	2.9 (-5)	2.2 (-5)
1.9 (-5)	3.10 (-5)	1.8 (-5)	3.30 (-5)	3.20 (-5)	1.8 (-5)	2.3 (-5)	1.8 (-5)
1.1 (-5)	2.65 (-5)	1.1 (-5)	2.95 (-5)	2.80 (-5)	1.1 (-5)	1.4 (-5)	1.1 (-5)
9.5 (-6)	1.38 (-5)	8.3 (-6)	1.43 (-5)	1.41 (-5)	8.7 (-6)	1.1 (-5)	8.4 (-6)
8.2 (-6)	1.20 (-5)	7.2 (-6)	1.24 (-5)	1.22 (-5)	7.5 (-6)	9.4 (-6)	7.3 (-6)
4.1 (-6)	6.30 (-6)	3.8 (-6)	6.60 (-6)	6.50 (-6)	3.9 (-6)	4.8 (-6)	3.7 (-6)
2.4 (-6)	5.30 (-6)	3.2 (-6)	5.60 (-6)	5.50 (-6)	3.3 (-6)	4.0 (-6)	3.2 (-6)
2.5 (-6)	4.00 (-6)	2.4 (-6)	4.20 (-6)	4.10 (-6)	2.4 (-6)	3.0 (-6)	2.3 (-6)
1.8 (-6)	2.95 (-6)	1.7 (-6)	3.00 (-6)	3.98 (-6)	1.7 (-6)	2.1 (-6)	1.7 (-6)
9.2 (-7)	1.32 (-6)	8.4 (-7)	1.46 (-6)	1.39 (-6)	8.4 (-7)	1.1 (-6)	8.6 (-7)
7.6 (-7)	1.10 (-6)	6.9 (-7)	1.21 (-6)	1.16 (-6)	7.0 (-7)	8.9 (-7)	7.2 (-7)
5.9 (-7)	8.70 (-7)	5.5 (-7)	9.70 (-7)	9.20 (-7)	5.5 (-7)	6.9 (-7)	5.7 (-7)
4.6 (-7)	6.90 (-7)	4.4 (-7)	7.90 (-7)	7.40 (-7)	4.3 (-7)	5.5 (-7)	4.5 (-7)
3.3 (-7)	5.10 (-7)	3.2 (-7)	5.80 (-7)	5.50 (-7)	3.2 (-7)	4.0 (-7)	3.3 (-7)
2.3 (-7)	3.65 (-7)	2.3 (-7)	4.25 (-7)	3.95 (-7)	2.2 (-7)	2.8 (-7)	2.3 (-7)

Note: Pressures are in torr. Numbers in parentheses are powers of 10.

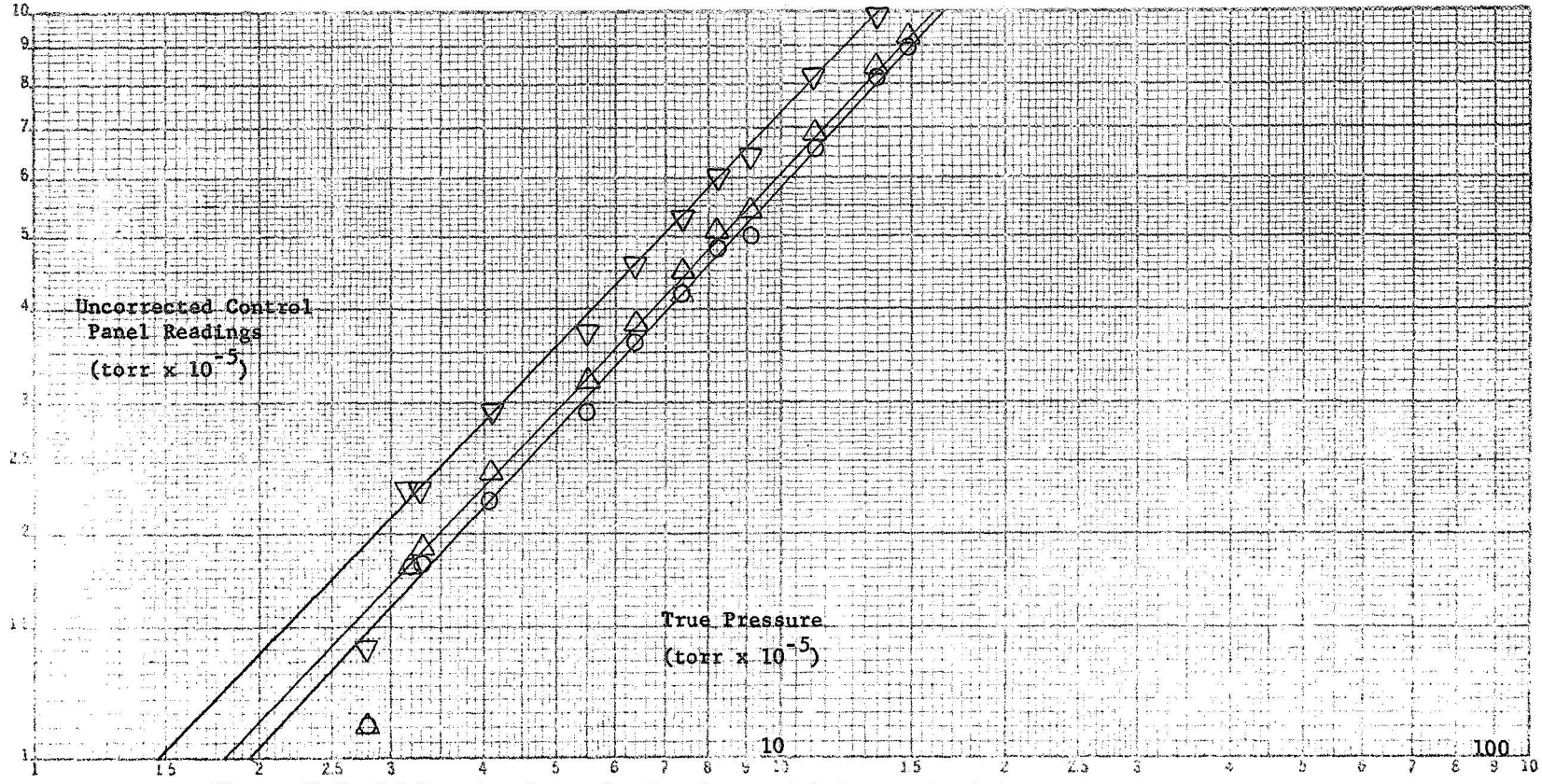


Figure 11.8 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. Third Set of Gages, 10^{-5} torr range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel #50004

○ Sample Ion Gage (P_1) with G.E. Control Panel #40012 Emission Current: 1.0 mA

△ Quartz Ion Gage (P_3) with G.E. Control Panel #40011

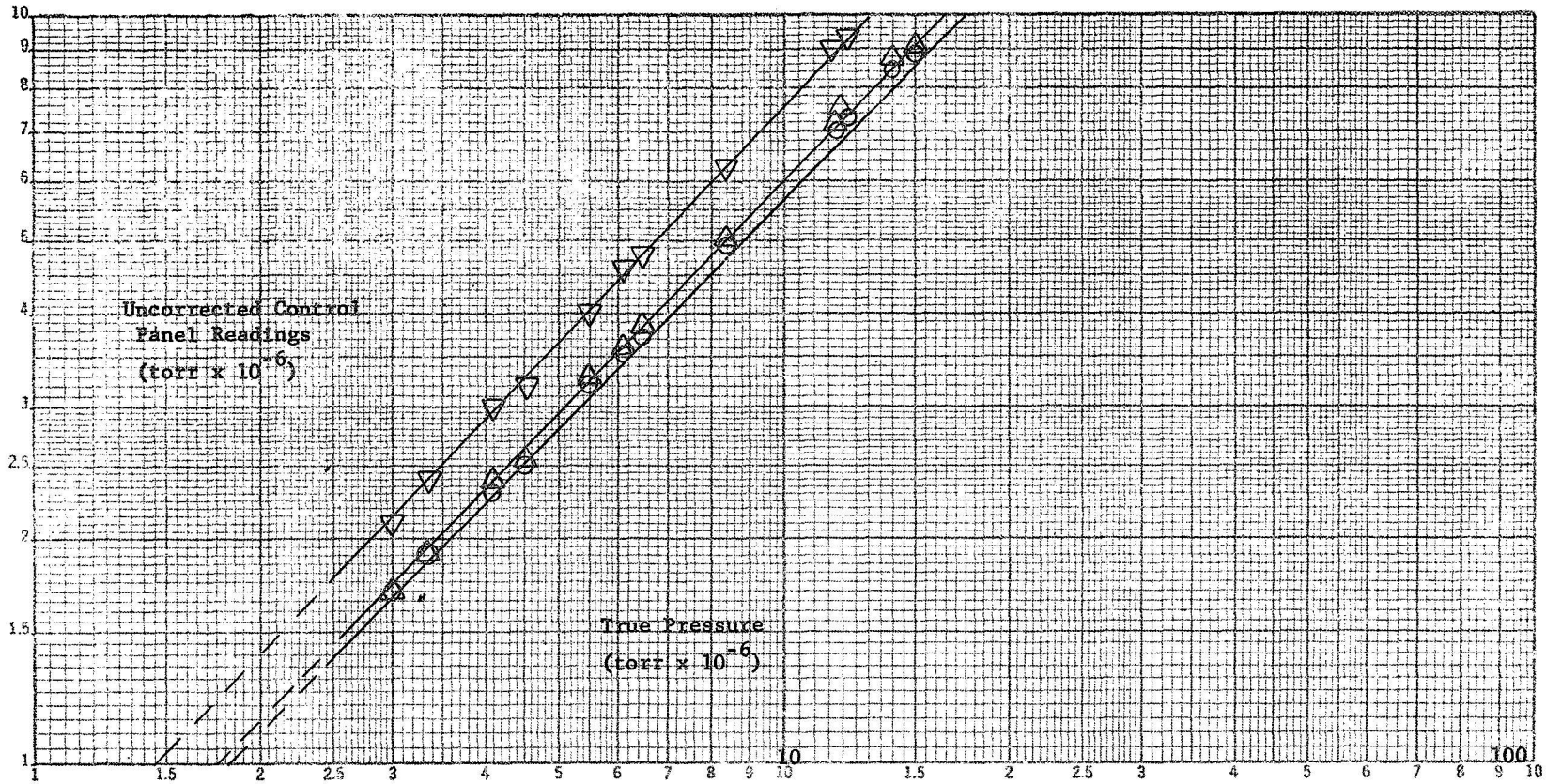


Figure 11.9 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. Third Set of Gages, 10^{-6} torr range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel #50004

○ Sample Ion Gage (P_1) with G.E. Control Panel #40012 Emission Current: 1.0 mA

△ Quartz Ion Gage (P_3) with G.E. Control Panel #40011

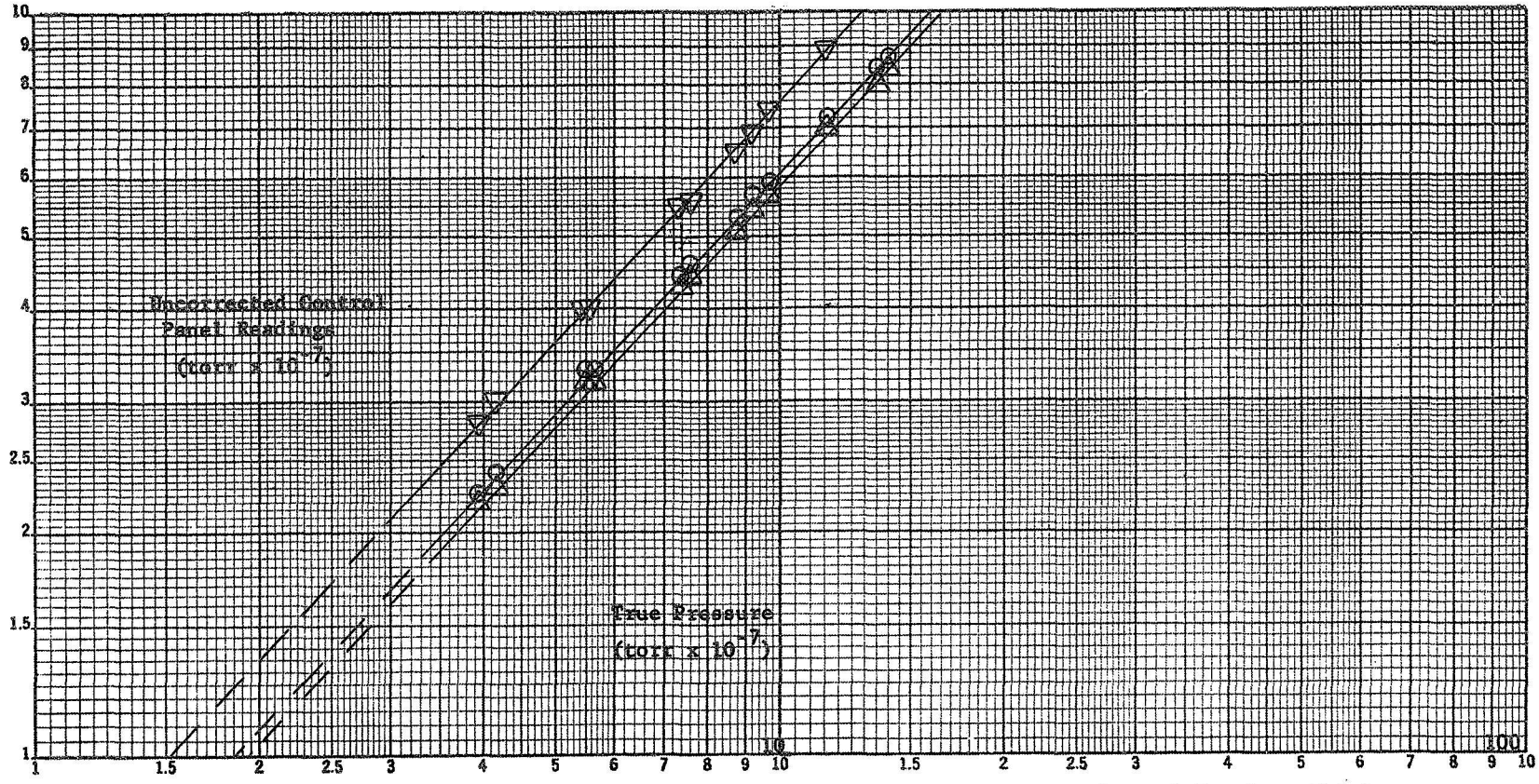


Figure 11.10 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. Third Set of Gages, 10^{-7} Torr Range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel #50004

○ Sample Ion Gage (P_1) with G.E. Control Panel #40012

△ Quartz Ion Gage (P_3) with G.E. Control Panel #40011 Emission Current: 1.0 mA

TABLE 11.7

Fourth Set of Ionization Gages

CALIBRATION OF 31 MAY 1967

#10 Indicated	#10 True AVCO Cal.	#11 Indicated	#11 True AVCO Cal	Avg. True	Quartz Indicated	Pump Indicated	Sample Indicated
8.7 (-5)	1.3 (-4)	7.7 (-5)	1.4 (-4)	1.35 (-4)	1.1 (-4)	9.6 (-5)	9.4 (-5)
7.1 (-5)	1.06 (-4)	6.4 (-5)	1.17 (-4)	1.11 (-4)	9.5 (-5)	7.7 (-5)	7.6 (-5)
5.1 (-5)	7.8 (-5)	4.6 (-5)	8.4 (-5)	8.1 (-5)	6.9 (-5)	5.5 (-5)	5.5 (-5)
4.2 (-5)	6.5 (-5)	3.8 (-5)	6.9 (-5)	6.70 (-5)	5.6 (-5)	4.5 (-5)	4.5 (-5)
3.2 (-5)	5.05 (-5)	3.0 (-5)	5.5 (-5)	5.28 (-5)	4.3 (-5)	3.5 (-5)	3.6 (-5)
2.4 (-5)	3.9 (-5)	2.2 (-5)	4.05 (-5)	3.97 (-5)	3.2 (-5)	2.7 (-5)	2.7 (-5)
1.7 (-5)	2.8 (-5)	1.4 (-5)	2.58 (-5)	2.69 (-5)	2.1 (-5)	1.8 (-5)	1.7 (-5)
8.4 (-6)	1.23 (-5)	7.2 (-6)	1.25 (-5)	1.24 (-5)	1.1 (-5)	8.7 (-6)	9.2 (-6)
7.0 (-6)	1.04 (-5)	6.1 (-6)	1.05 (-5)	1.045(-5)	8.9 (-6)	7.4 (-6)	7.7 (-6)
6.1 (-6)	9.0 (-6)	5.4 (-6)	9.3 (-6)	9.15 (-6)	7.8 (-6)	6.4 (-6)	6.8 (-6)
5.0 (-6)	7.6 (-6)	4.4 (-6)	7.6 (-6)	7.6 (-6)	6.4 (-6)	5.3 (-6)	5.5 (-6)
3.1 (-6)	4.9 (-6)	2.8 (-6)	4.4 (-6)	4.65 (-6)	4.0 (-6)	3.3 (-6)	3.5 (-6)
2.4 (-6)	3.8 (-6)	2.2 (-6)	3.85 (-6)	3.83 (-6)	3.1 (-6)	2.6 (-6)	2.7 (-6)
1.8 (-6)	2.95 (-6)	1.7 (-6)	3.0 (-6)	2.98 (-6)	2.4 (-6)	2.0 (-6)	2.1 (-6)
1.6 (-6)	2.6 (-6)	1.4 (-6)	2.45 (-6)	2.53 (-6)	2.0 (-6)	1.7 (-6)	1.75(-6)
8.4 (-7)	1.2 (-6)	7.4 (-7)	1.3 (-6)	1.25 (-6)	1.1 (-6)	9.2 (-7)	9.3 (-7)
7.5 (-7)	1.08 (-6)	6.7 (-7)	1.17 (-6)	1.13 (-6)	9.4 (-7)	8.2 (-7)	8.3 (-7)
6.8 (-7)	9.9 (-7)	6.1 (-7)	1.07 (-6)	9.99 (-7)	8.5 (-7)	7.4 (-7)	7.5 (-7)
5.4 (-7)	8.0 (-7)	5.0 (-7)	8.9 (-7)	8.45 (-7)	6.9 (-7)	6.0 (-7)	6.2 (-7)
4.6 (-7)	6.9 (-7)	4.2 (-7)	7.5 (-7)	7.2 (-7)	5.9 (-7)	5.0 (-7)	5.3 (-7)
4.0 (-7)	6.1 (-7)	3.8 (-7)	6.9 (-7)	6.5 (-7)	5.2 (-7)	4.5 (-7)	4.6 (-7)

Note: Pressures are in torr. Numbers in parentheses are powers of 10.

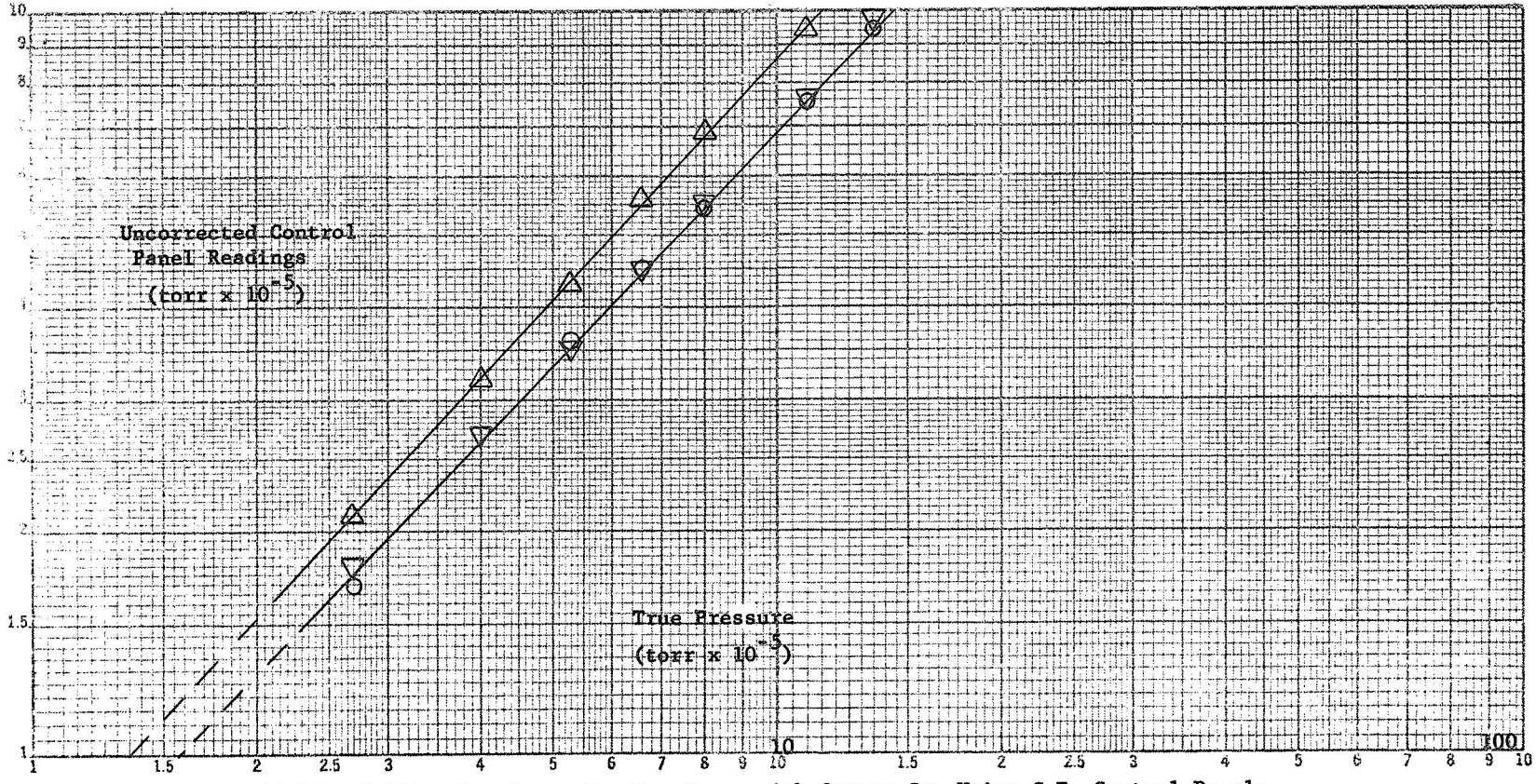


Figure 11.11 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. Fourth Set of Gages, 10^{-5} Torr Range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel #50004

○ Sample Ion Gage (P_1) with G.E. Control Panel #40012

△ Quartz Ion Gage (P_3) with G.E. Control Panel #40011 Emission Current: 1.0 mA

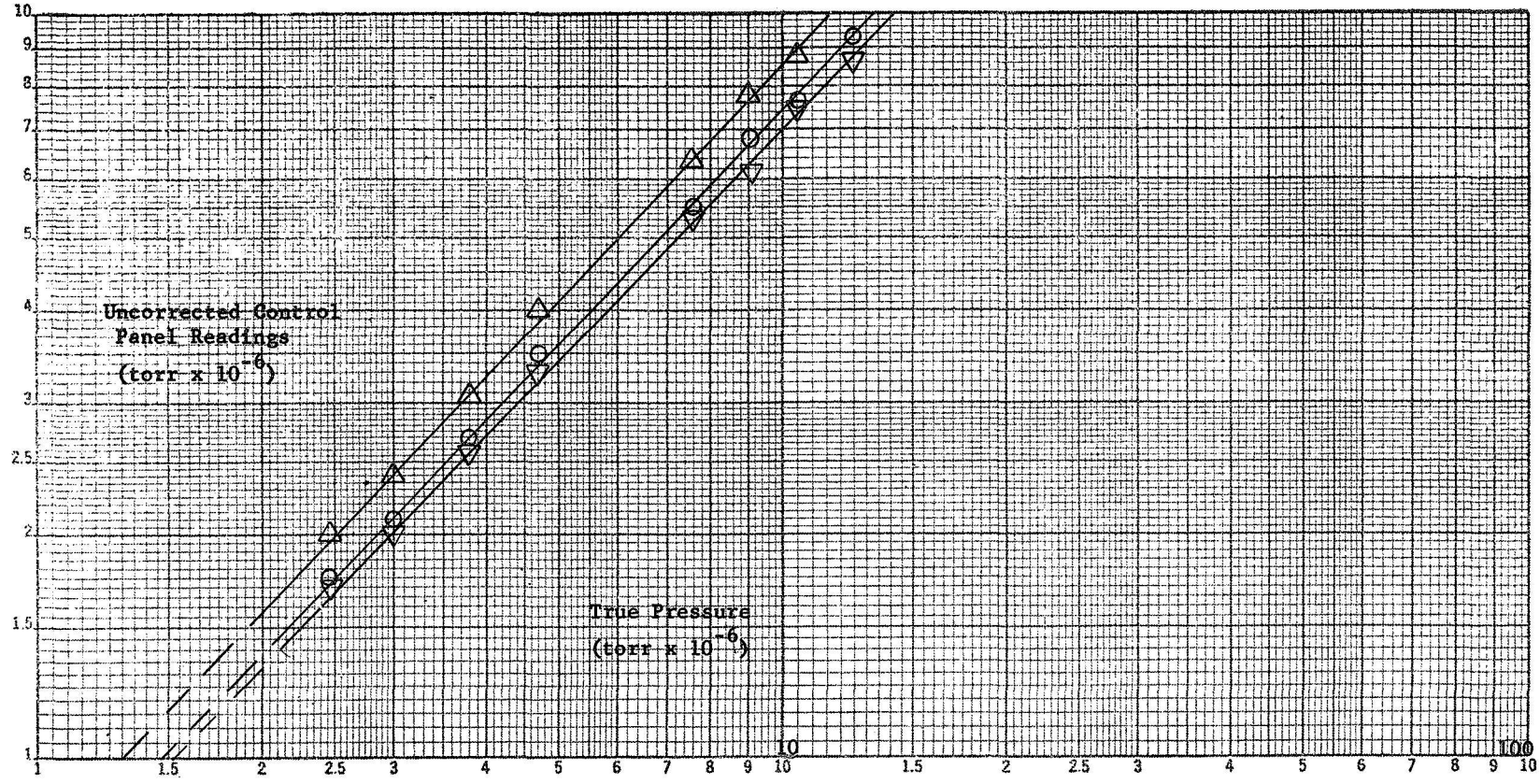


Figure 11.12 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. Fourth Set of Gages, 10^{-6} Torr Range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel #50004

○ Sample Ion Gage (P_1) with G.E. Control Panel #40012

△ Quartz Ion Gage (P_3) with G.E. Control Panel #40011 Emission Current: 1.0 mA

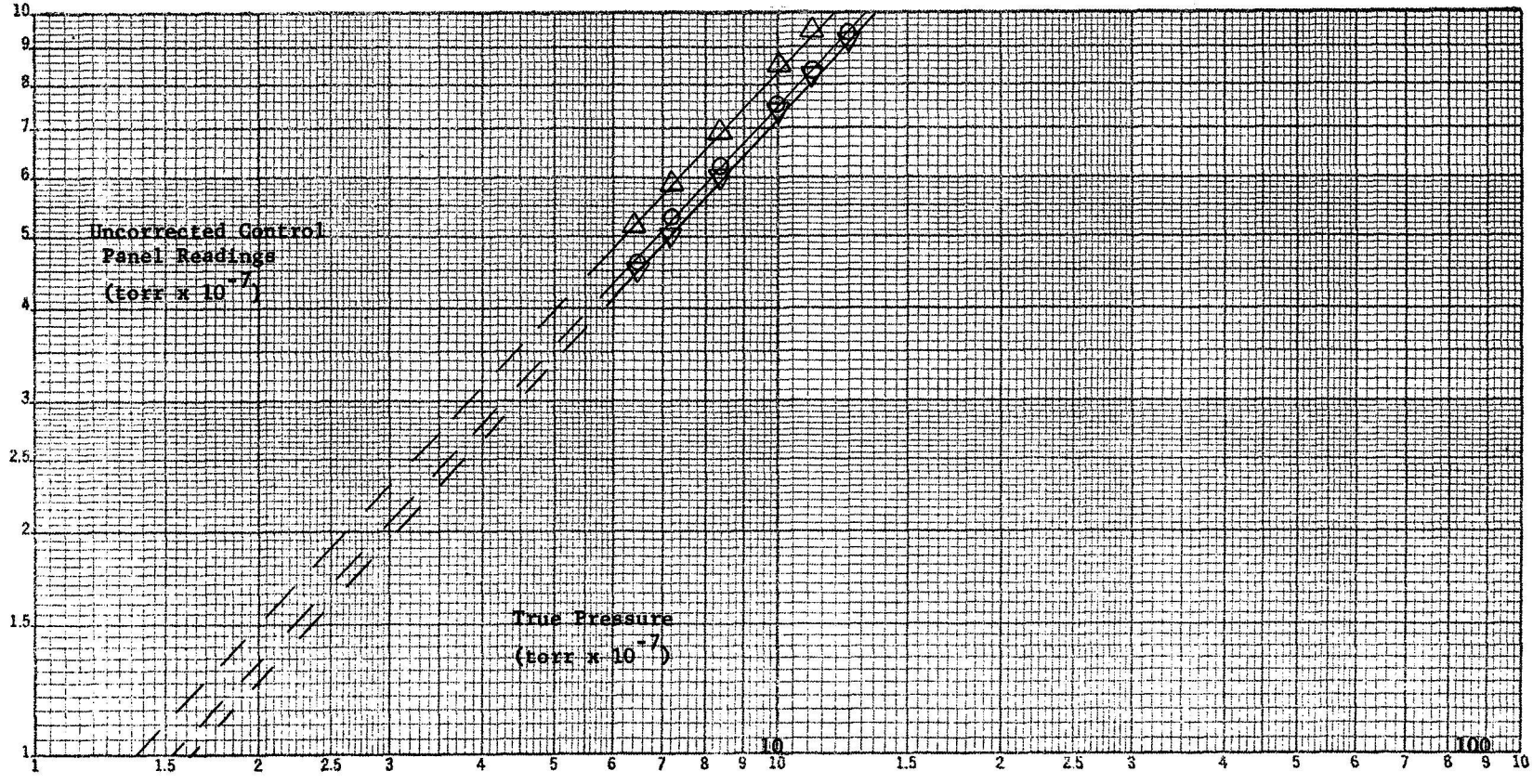


Figure 11.13 Calibration Curve for Ion Gages with Oxygen Gas Using G.E. Control Panels. Fourth Set of Gages, 10^{-7} Torr Range.

▽ Pump Ion Gage (P_o) with G.E. Control Panel #50004

○ Sample Ion Gage (P_1) with G.E. Control Panel #40012

△ Quartz Ion Gage (P_3) with G.E. Control Panel #40011 Emission Current: 1.0 mA

11.2 Calibrations of Partial Pressure Analyzers

Two sets of partial pressure analyzer calibrations were taken during the program. These calibrations were run in order to establish sensitivity values for each gas of interest with a particular mass spectrometer. These values were needed to correct peak height readings of the partial pressure analyzers into percent values of the gas compositions. These in turn were used to correct ionization gage readings at each of the three locations.

First Set of Partial Pressure Analyzer Calibrations

The three partial pressure analyzers were calibrated in the following manner. Five gases - CO, O_2, H_2, CO_2, and N_2 - were introduced into the vacuum system through a 10^{-7} and cc/sec leak which had a Δp of approximately 1 atmosphere. The pressure in the system before introduction of the gases was in the middle of the 10^{-7} range. Vacuum was applied on the 1 atm. side of the leak with a sorption pump. At this time, background readings of the particular gas of interest were taken. After the background readings, the particular gas of interest was backfilled to 1 atm on the leak. When steady state conditions were reached, the peak height of the particular gas of interest was taken. The total reading being the peak height with the gas present in the system minus the background peak height. This procedure was followed for each of the gases mentioned above in the order that they are listed. A run consisted of calibrations for all five gases. Six runs were made and the data is listed on Tables 11.8 and 11.9. Plots of the ratios of the various gases to H_2 versus runs are given in Figures 11.14, 11.15, and 11.16. The three partial pressure analyzers are identified by their location, namely, "P" for P_0 location, "S" for P_1 location and "Q" for the P_3 location.

TABLE 11.8

FIRST SET:

CALIBRATION OF PARTIAL PRESSURE ANALYZERS

	1st Run	2nd Run	3rd Run	4th Run	5th Run	6th Run
	Q	Q	Q	Q	Q	Q
CO	2.080	1.880	1.480	1.390	1.280	1.140
O_2	0.800	0.659	0.575	0.564	0.465	0.400
H_2	2.830	2.350	2.210	1.860	1.830	1.720
CO_2	1.180	0.915	0.872	0.766	0.716	0.690
N_2	1.480	1.470	1.280	1.290	1.180	1.140
	P	P	P	P	P	P
CO	1.070	1.070	0.939	0.949	0.888	0.949
O_2	0.319	0.320	0.296	0.307	0.284	0.274
H_2	6.900	6.430	6.230	5.900	6.070	5.770
CO_2	0.156	0.164	0.155	0.157	0.137	0.144
N_2	0.859	0.924	0.968	0.911	0.975	0.826
	S	S	S	S	S	S
CO	0.730	0.75ρ	0.705	0.684	0.686	0.652
O_2	0.292	0.262	0.277	0.229	0.232	0.181
H_2	4.180	3.380	3.400	2.850	2.480	2.200
CO_2	0.434	0.410	0.387	0.368	0.302	0.330
N_2	0.620	0.705	0.631	0.733	0.628	0.545

NOTE: The designations Q, P, and S represent the locations of the mass spectrometers. All data is in amperes (x 10^{-7}).

SENSITIVITY AVERAGES

	Q	P	S
H_2	2.13	6.22	3.08
O_2	0.577	0.300	0.246
CO	1.54	0.978	0.702
CO_2	0.857	0.152	0.372
N_2	1.31	0.906	0.644

TABLE 11.9

FIRST SET:PARTIAL PRESSURE ANALYZER CALIBRATIONS

RATIO DATA USING H_2 AS REFERENCE

	Q_{first}	Q_{second}	Q_{third}	Q_{fourth}	Q_{fifth}	Q_{sixth}
CO	0.735	0.800	0.670	0.750	0.700	0.662
O_2	0.283	0.280	0.260	0.302	0.178	0.232
CO_2	0.417	0.390	0.394	0.412	0.392	0.400
N_2	0.522	0.625	0.579	0.691	0.645	0.662

	P_{first}	P_{second}	P_{third}	P_{fourth}	P_{fifth}	P_{sixth}
CO	0.155	0.166	0.150	0.160	0.146	0.164
O_2	0.0464	0.0497	0.0475	0.0520	0.0467	0.0475
CO_2	0.0226	0.0256	0.0249	0.0266	0.0226	0.0249
N_2	0.124	0.144	0.155	0.154	0.160	0.143

	S_{first}	S_{second}	S_{third}	S_{fourth}	S_{fifth}	S_{sixth}
CO	0.175	0.223	0.207	0.240	0.256	0.296
O_2	0.0700	0.0775	0.815	0.0805	0.0935	0.0822
CO_2	0.108	0.121	0.114	0.129	0.113	0.145
N_2	0.149	0.208	0.186	0.257	0.253	0.248

Figure 11.14 First Set of Calibration Data for "P" Partial Pressure Analyzer

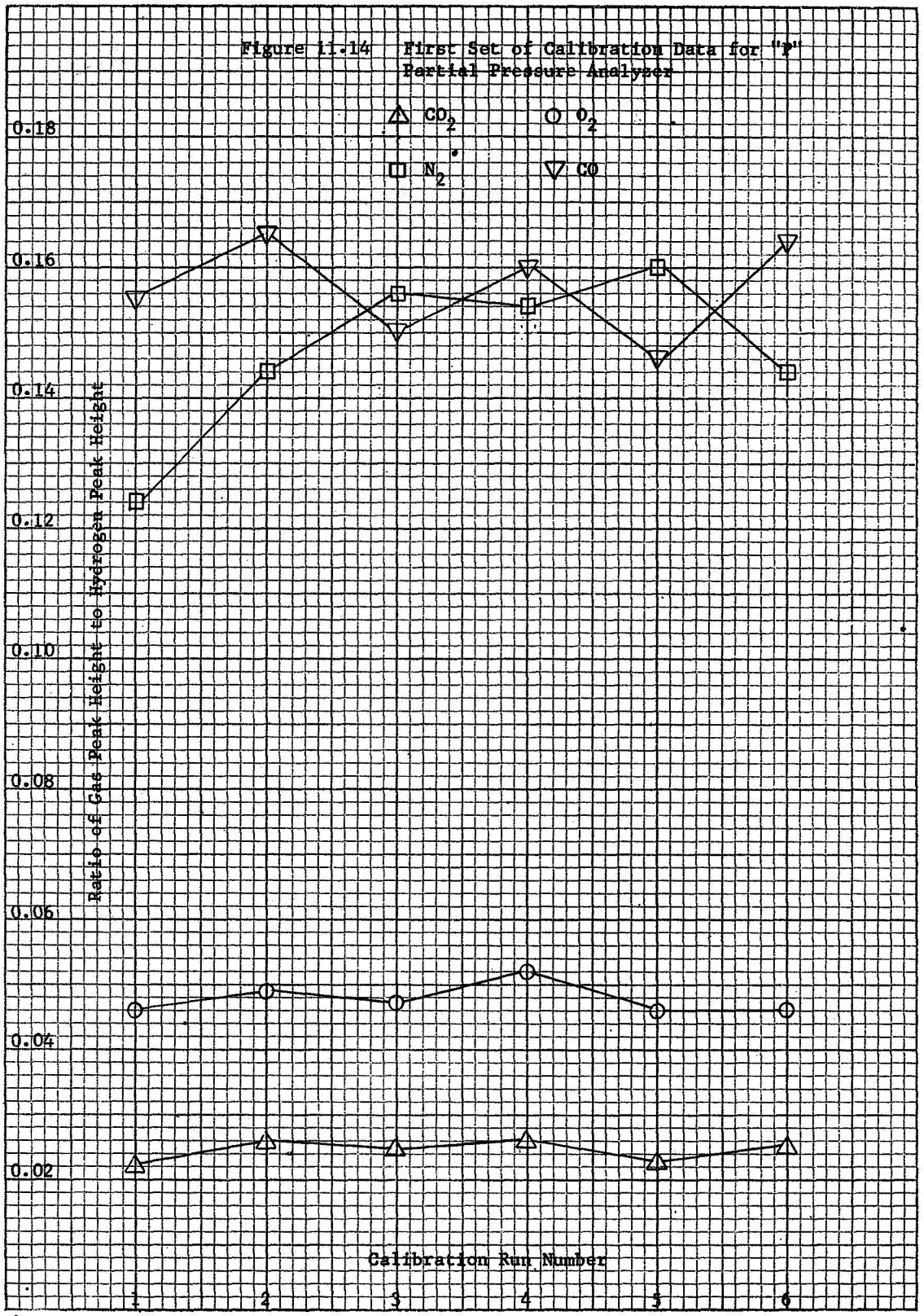


Figure 11.15 First Set of Calibration Data for "S" Partial Pressure Analyzer

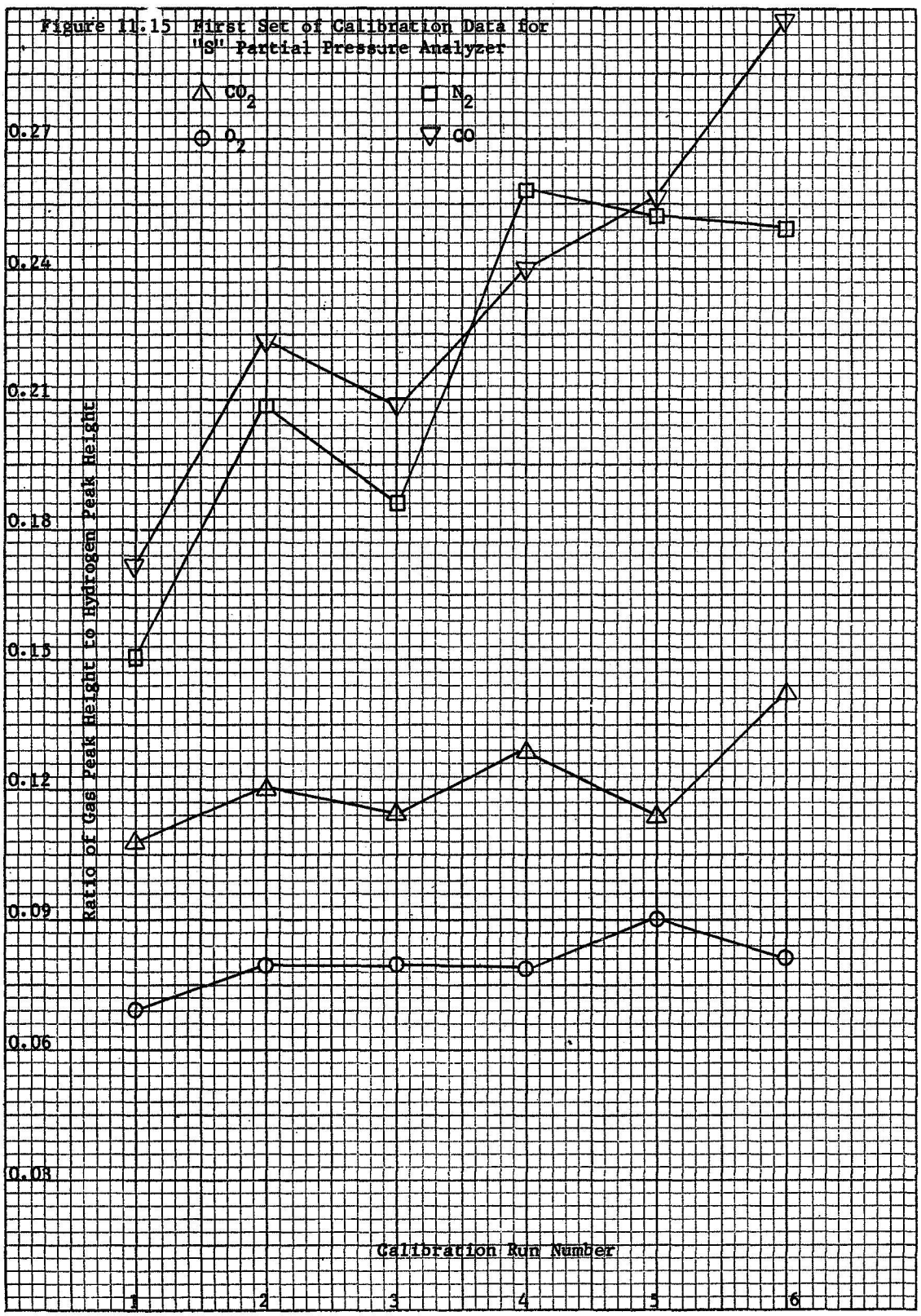


Figure 11.16 First Set of Calibration Data for "Q" Partial Pressure Analyzer

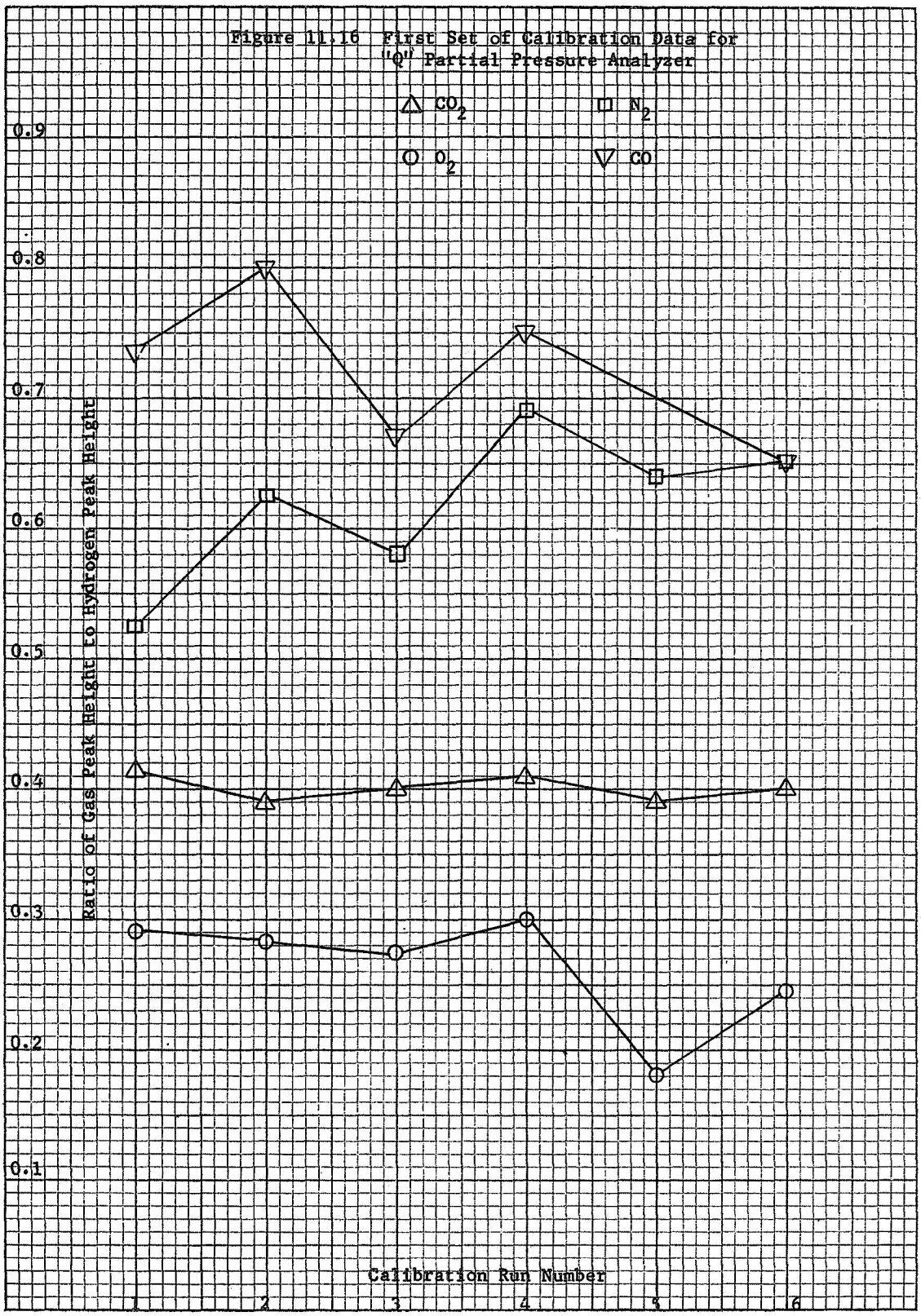


Figure 11.17 is a plot of H_2 peak height versus runs. The average values obtained for each of the gases will be used as a sensitivity factor in determining percent values of sample readings during the tests.

In this set the sensitivities for H_2O and He were assumed to be 1.00 and 0.85 respectively.

Example of Percent Composition Calculations

The following is data taken from Run # 1 using the first set of partial pressure analyzers. The data was taken from peak height plots on the strip chart recorders and tabulated below. Using sensitivity values from Table 11.8, percent compositions were arrived at for each of the mass spectrometers.

Recorder Readings

	"Q"	"S"	"P"
CO_2	.329	-	-
O_2	.681	.288	1.34
CO	1.26	.078	-
H_2O	.514	.018	-
16	0.70	0.45	0.45
He	.032	.028	-
H_2	.694	.416	.17

Percent Values

	"S" P.P.A.		% Values
$\frac{O_2}{\text{Sensitivity}}$	$= \frac{.288}{0.246} =$	1.170	$O_2 = 80.80$
$\frac{CO}{\text{Sensitivity}}$	$= \frac{.078}{0.978} =$	.080	CO = 5.51
$\frac{H_2}{\text{Sensitivity}}$	$= \frac{.416}{3.08} =$	.135	$H_2 = 9.32$
$\frac{H_2O}{\text{Sensitivity}}$	$= \frac{.018}{1.00} =$	.018	$H_2O = 1.23$
$\frac{C_O2}{\text{Sensitivity}}$	$= \frac{.005}{.372} =$	.013	$C_O2 = .90$
$\frac{He}{\text{Sensitivity}}$	$= \frac{.028}{.85} =$	.033	He = 2.27
		1.449	

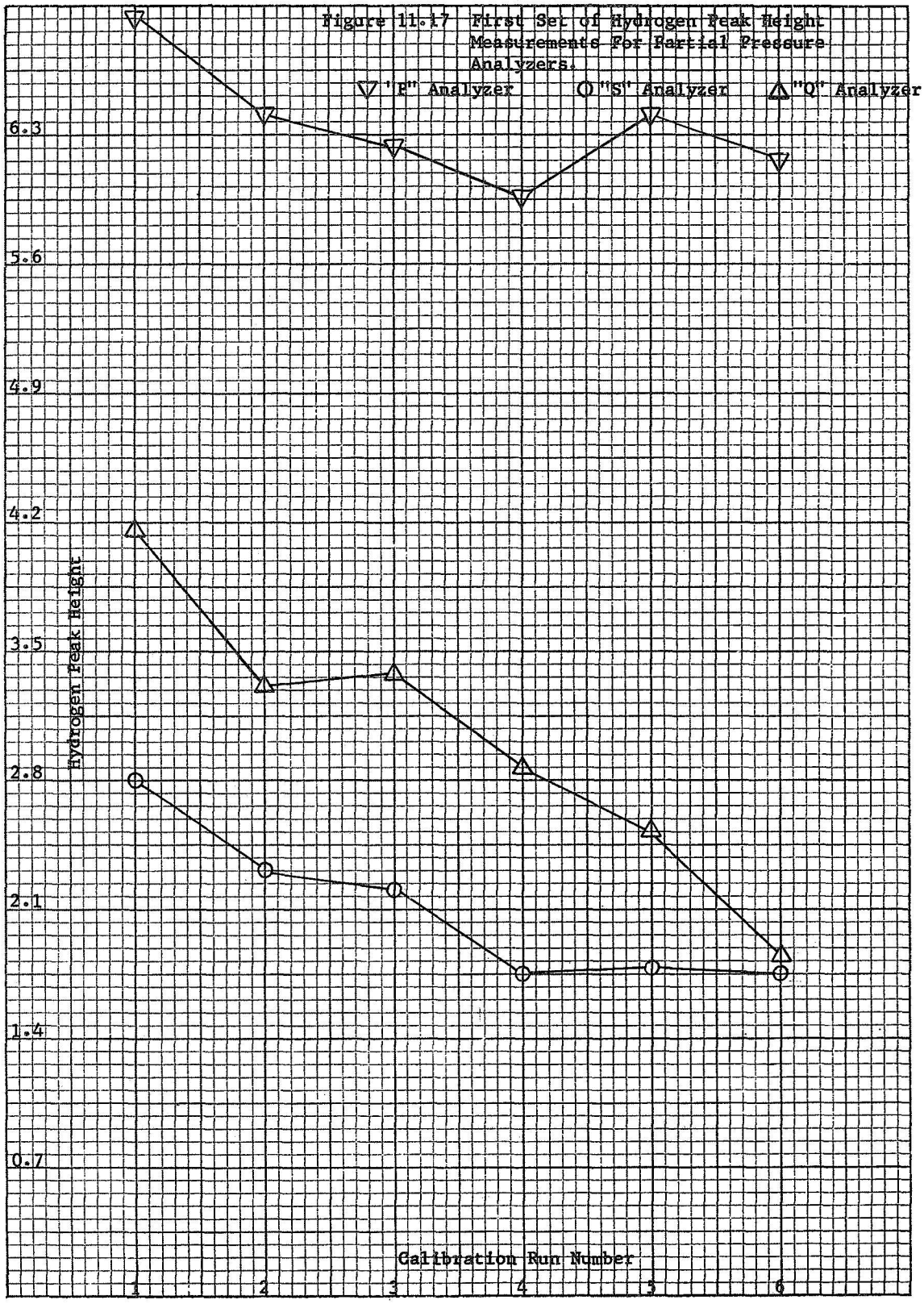
	"P" P.P.A.		% Values
$\frac{O_2}{\text{Sensitivity}}$	$= \frac{1.34}{0.300} =$	4.480	$O_2 = 99.35$
$\frac{H_2}{\text{Sensitivity}}$	$= \frac{.17}{6.22} =$	.027	$H_2 = .65$
		5.507	

	"Q" P.P.A.			% Values		
$\frac{CO_2}{\text{Sensitivity}}$	= $\frac{.329}{0.857}$	=	.384	CO_2	=	11.15
$\frac{O_2}{\text{Sensitivity}}$	= $\frac{.681}{0.577}$	=	1.18	O_2	=	33.83
$\frac{CO}{\text{Sensitivity}}$	= $\frac{1.26}{1.54}$	=	.816	CO	=	23.65
$\frac{H_2O}{\text{Sensitivity}}$	= $\frac{.514}{1.00}$	=	.514	H_2O	=	14.93
$\frac{H_2}{\text{Sensitivity}}$	= $\frac{.694}{2.13}$	=	.326	H_2	=	15.24
$\frac{He}{\text{Sensitivity}}$	= $\frac{.032}{.85}$	=	$\frac{.037}{3.257}$	He	=	1.20

Second Set of Partial Pressure Analyzer Calibrations

The three partial pressure analyzers were calibrated in the following manner. Six gases - CO, O_2, H_2, CO_2, N_2 and H_2O vapor were introduced into the vacuum system through a 3.1×10^{-3} std cc/sec leak which had a Δp of approximately 4 torr. The pressure in the system before introduction of the gases was 1×10^{-6} range. Vacuum was applied on the 4 torr side of the leak with a sorption pump. At this time, background readings of the particular gas of interest were taken. After the background readings, the particular gas of interest was backfilled to 4 torr on the leak. When steady state conditions were reached, the peak height of the particular gas of interest was taken. The total reading being the peak height with the gas present in the system minus the background peak height. This procedure was followed for each of the gases mentioned above in the order that they are listed. A run consisted of

Figure 11.17 First Set of Hydrogen Peak Height Measurements For Partial Pressure Analyzers.



calibration for all six gases. Three runs were made and the data is listed on Tables 11.10 and 11.11. Plots of the ratios of the various gases to H_2 versus runs are given in Figures 11.18, 11.19 and 11.20. The three partial pressure analyzers are identified by their location, namely, "P" for P_0 location, "S" for P_1 location and "Q" for the P_3 location. Figure 11.21 is a plot of H_2 peak height versus runs. The average values obtained for each of the gases were used as a sensitivity factor in determining percent values of sample readings during the tests.

TABLE 11.10

SECOND SET: CALIBRATION OF PARTIAL PRESSURE ANALYZERS

	First Run Q	Second Run Q	Third Run Q	First Run S	Second Run S	Third Run S	First Run P	Second Run P	Third Run P
CO	1.45	1.66	0.723	0.0616	0.0583	0.0474	0.288	0.289	0.231
O_2	0.600	0.616	0.364	0.0227	0.022	0.0200	0.131	0.133	0.105
H_2	1.24	1.16	0.887	0.326	0.377	0.297	0.248	0.234	0.231
CO_2	0.497	0.367	0.297	0.0281	0.0223	0.0194	0.171	0.165	0.146
N_2	0.96	0.893	0.723	0.0637	0.0514	0.0456	0.280	0.270	0.232
H_2O	0.253	0.200	0.145	0.0185	0.0137	0.0116	0.078	0.0606	0.0558

NOTE: The designations Q, P, and S represent the locations of the mass spectrometers. All data is in microamps.

SENSITIVITY AVERAGES

	Q	S	P
H_2	1.092	0.334	0.237
O_2	0.528	0.0216	0.123
CO	1.278	0.0558	0.269
CO_2	0.387	0.0233	0.161
N_2	0.859	0.0536	0.261
H_2O	0.199	0.0146	0.0648

TABLE 11.11

SECOND SET: PARTIAL PRESSURE ANALYZER CALIBRATIONS

RATIO DATA USING H_2 AS REFERENCE

	Q First	Q Second	Q Third
CO	1.17	1.43	0.815
O_2	0.487	0.531	0.411
CO_2	0.400	0.316	0.335
N_2	0.775	0.770	0.815
H_2O	0.205	0.172	0.164
	S	S	S
CO	0.189	0.155	0.159
O_2	0.0696	0.0584	0.0673
CO_2	0.0862	0.0591	0.0654
N_2	0.195	0.136	0.154
H_2O	0.0568	0.0364	0.039
	P	P	P
CO	1.28	1.23	1.00
O_2	0.528	0.570	0.455
CO_2	0.690	0.704	0.633
N_2	1.13	1.15	1.00
H_2O	0.314	0.259	0.241

Figure 11.18 Second Set of Calibration Data For "P" Partial Pressure Analyzer

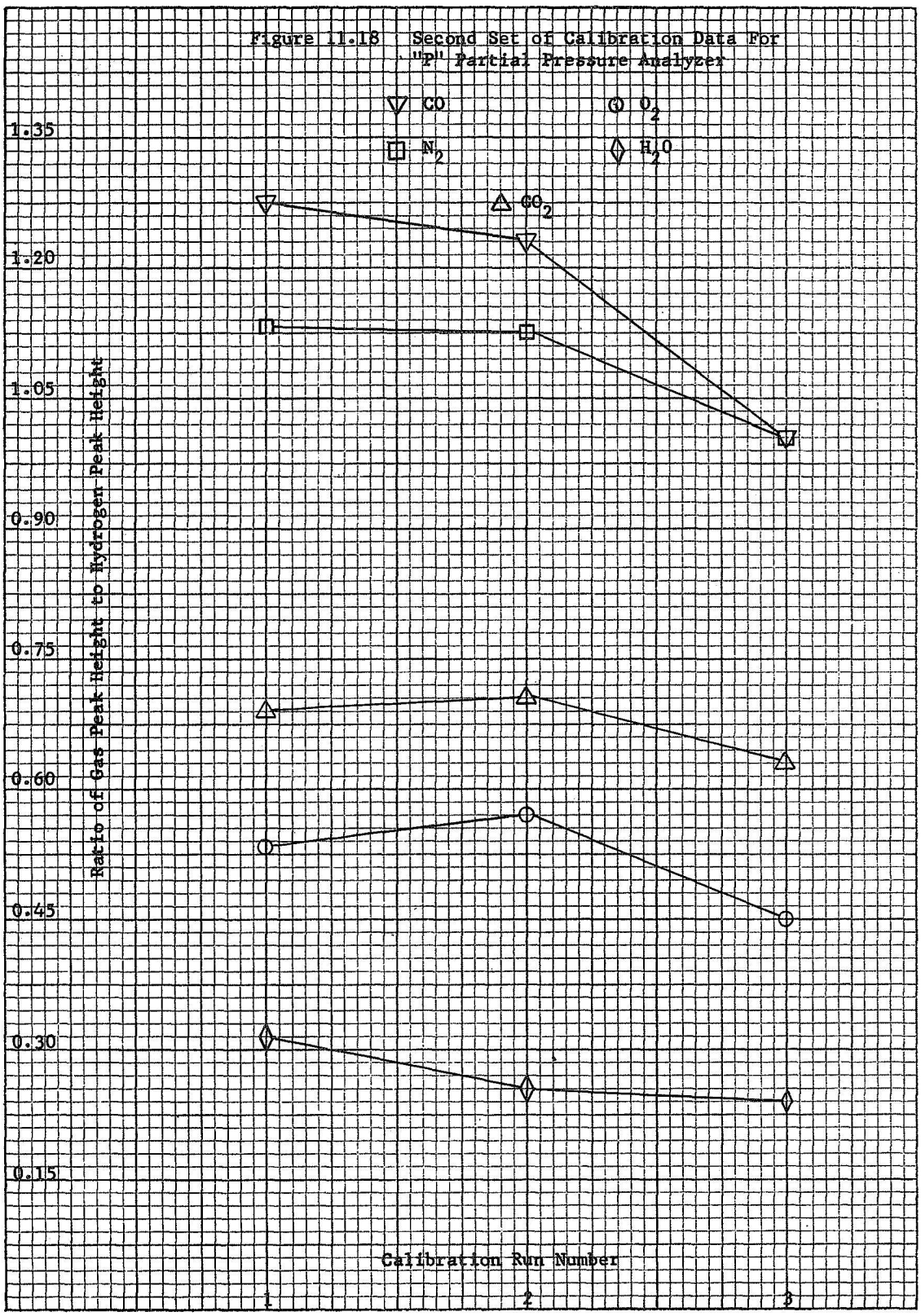


Figure 11.19 Second Set of Calibration Data For "S" Partial Pressure Analyzer

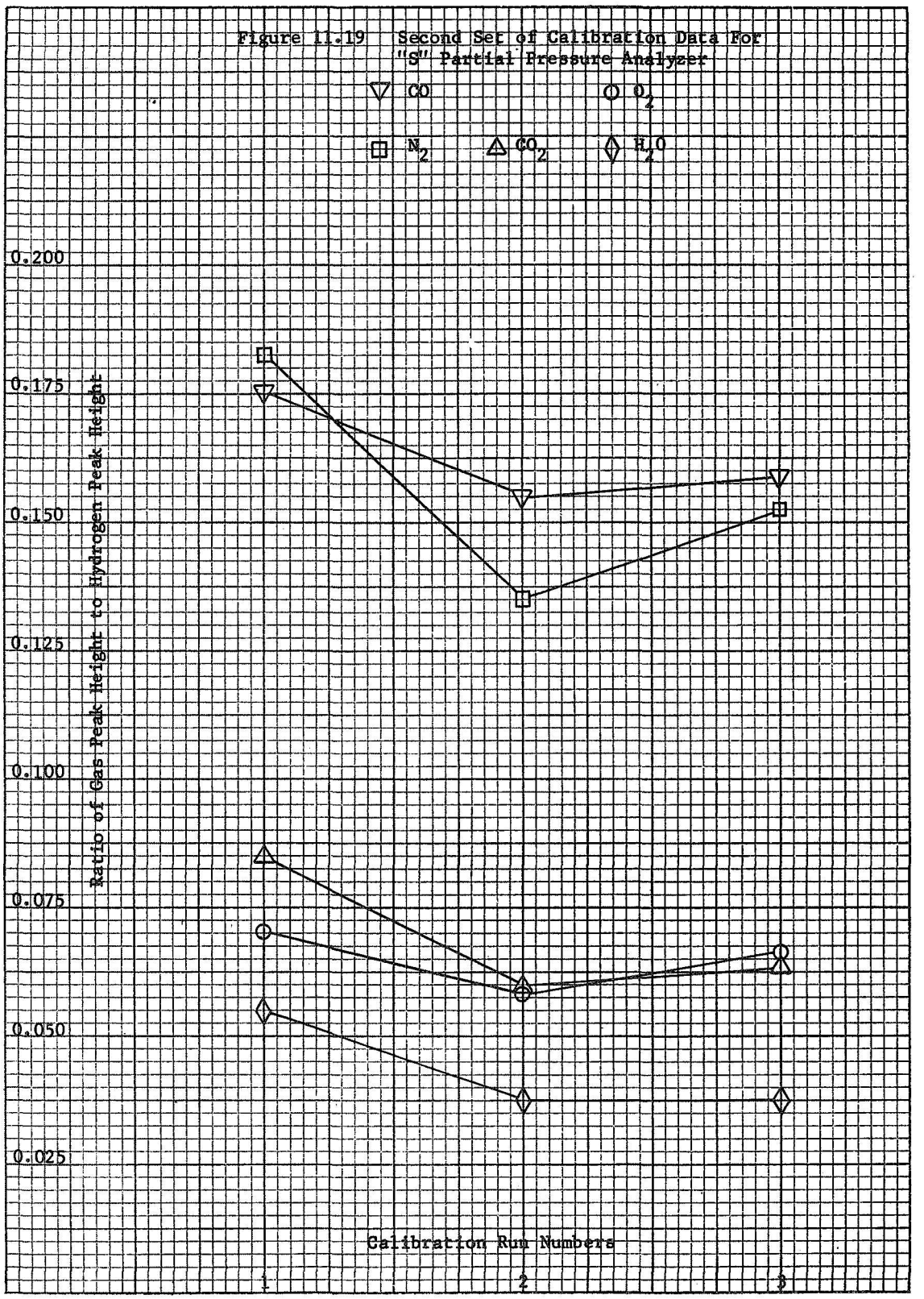


Figure 11.20 Second Set of Calibration Data for "Q" Partial Pressure Analyzer

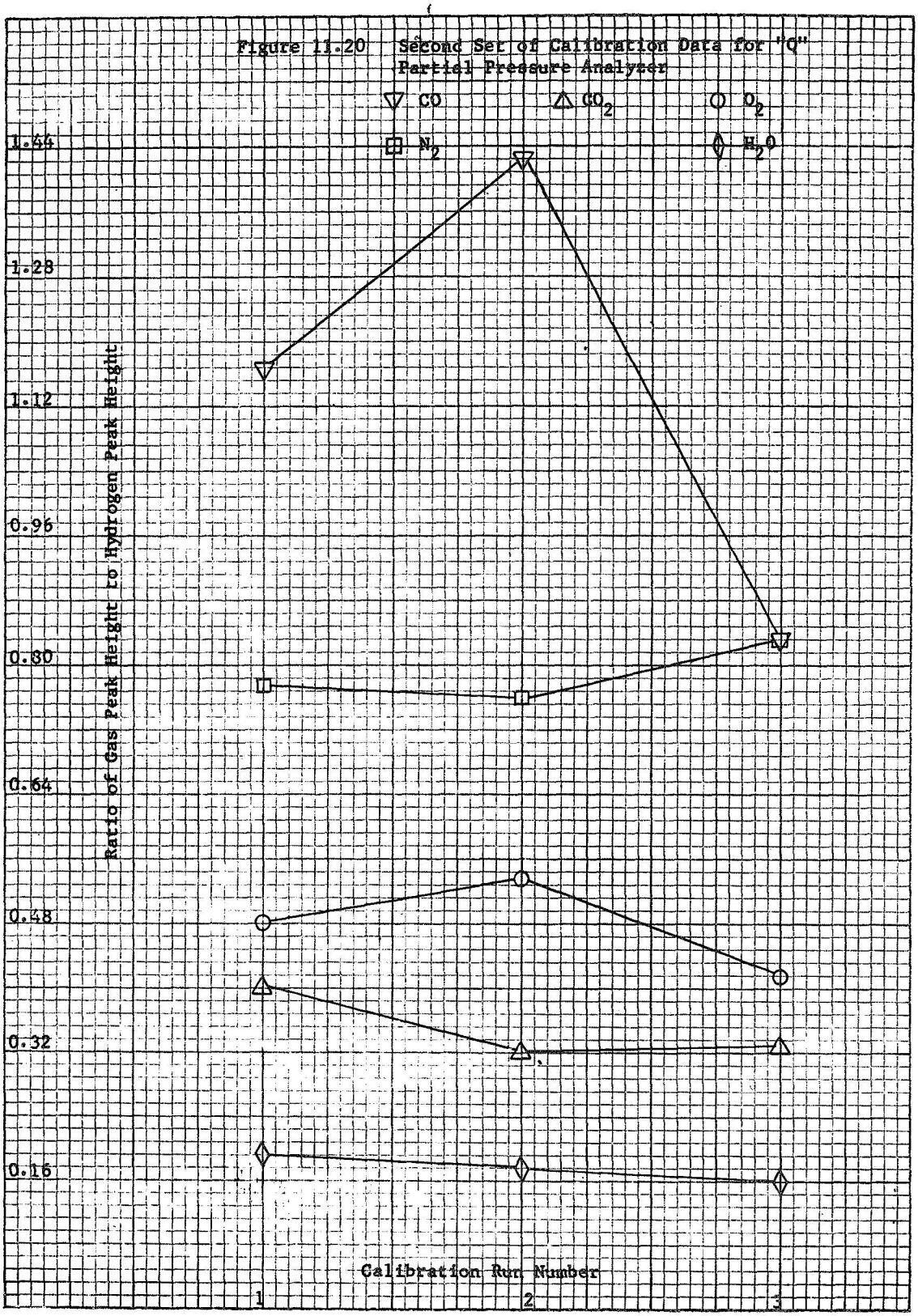
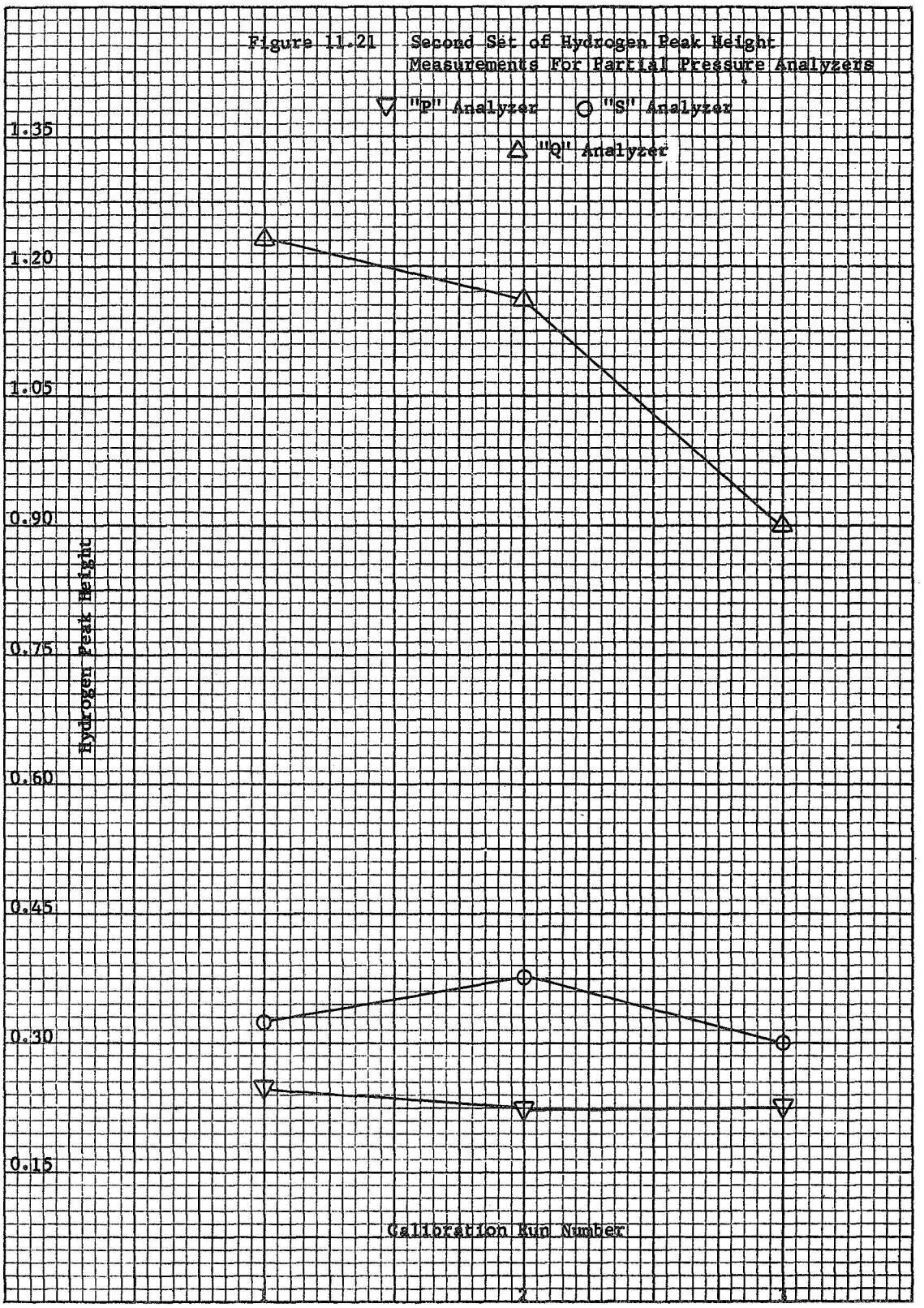


Figure 11.21 Second Set of Hydrogen Peak Height Measurements For Partial Pressure Analyzers



Ionization Gage Data During Calibration of Second Set of Partial Pressure Analyzers

It was felt that recording ionization gage data during the mass spectrometer calibration might be helpful in determining sensitivity values for the ionization gages. The following is the data collected during the exposure of the system to CO, O_2, H_2, CO_2, N_2 and water vapor and is presented in Tables 11.12, 11.13 and 11.14.

TABLE 11.12

IONIZATION GAGE DATA RECORDED DURING

CALIBRATION OF PARTIAL PRESSURE ANALYZERS

Run	Gas	Gage	Before	After	Δp
1	H_2	P	1.4(-6)	6.2(-6)	4.8(-6)
		S	2.0(-6)	8.9(-6)	6.9(-6)
		Q	2.0(-6)	8.6(-6)	6.6(-6)
2	H_2	P	1.2(-6)	6.2(-6)	5.0(-6)
		S	1.6(-6)	8.6(-6)	7.0(-6)
		Q	1.6(-6)	8.2(-6)	6.6(-6)
3	H_2	P	1.4(-6)	6.0(-6)	4.6(-6)
		S	1.9(-6)	8.5(-6)	6.6(-6)
		Q	1.9(-6)	8.2(-6)	6.3(-6)
1	CO_2	P	1.6(-6)	1.3(-5)	11.4(-6)
		S	2.2(-6)	1.8(-5)	15.8(-6)
		Q	2.2(-6)	1.8(-5)	15.8(-6)
2	CO_2	P	1.4(-6)	1.3(-5)	11.6(-6)
		S	1.7(-6)	1.8(-5)	16.3(-6)
		Q	1.7(-6)	1.8(-5)	16.3(-6)
3	CO_2	P	1.5(-6)	1.2(-5)	10.5(-6)
		S	2.0(-6)	1.7(-5)	15.0(-6)
		Q	1.9(-6)	1.7(-5)	15.1(-6)
1	N_2	P	1.5(-6)	1.0(-5)	8.5(-6)
		S	2.0(-6)	1.4(-5)	12.0(-6)
		Q	2.0(-6)	1.4(-5)	12.0(-6)
2	N_2	P	1.3(-6)	1.0(-5)	8.7(-6)
		S	1.6(-6)	1.4(-5)	12.4(-6)
		Q	1.6(-6)	1.4(-5)	12.4(-6)
3	N_2	P	1.3(-6)	1.0(-5)	8.7(-6)
		S	1.8(-6)	1.4(-5)	12.2(-6)
		Q	1.7(-6)	1.4(-5)	12.3(-6)

TABLE 11.13

IONIZATION GAGE DATA RECORDED DURING

CALIBRATION OF PARTIAL PRESSURE ANALYZERS

Run	Gas	Gage	Before	After	Δp
1	H_2O	P	1.3(-6)	5.6(-6)	4.3(-6)
		S	1.6(-6)	8.7(-6)	7.1(-6)
		Q	1.6(-6)	8.4(-6)	6.8(-6)
2	H_2O	P	1.0(-6)	5.3(-6)	4.3(-6)
		S	1.3(-6)	8.1(-6)	6.8(-6)
		Q	1.4(-6)	7.9(-6)	6.5(-6)
3	H_2O	P	1.1(-6)	6.5(-6)	5.4(-6)
		S	1.6(-6)	9.6(-6)	8.0(-6)
		Q	1.6(-6)	9.3(-6)	7.7(-6)
1	CO	P	1.4(-6)	1.1(-5)	9.6(-6)
		S	1.9(-6)	1.5(-5)	13.1(-6)
		Q	1.8(-6)	1.5(-5)	13.2(-6)
2	CO	P	1.1(-6)	1.0(-5)	8.9(-6)
		S	1.4(-6)	1.4(-5)	12.6(-6)
		Q	1.4(-6)	1.4(-5)	12.6(-6)
3	CO	P	1.5(-6)	1.0(-5)	8.5(-6)
		S	2.0(-6)	1.5(-5)	13.0(-6)
		Q	2.0(-6)	1.5(-5)	13.0(-6)
1	O_2	P	1.4(-6)	7.5(-6)	6.1(-6)
		S	1.9(-6)	1.1(-5)	9.1(-6)
		Q	1.9(-6)	1.0(-5)	8.1(-6)
2	O_2	P	1.4(-6)	7.1(-6)	5.7(-6)
		S	2.0(-6)	1.0(-5)	8.0(-6)
		Q	2.0(-6)	1.0(-5)	8.0(-6)
3	O_2	P	1.4(-6)	7.2(-6)	5.8(-6)
			1.8(-6)	1.0(-5)	8.2(-6)
			1.8(-6)	1.0(-5)	8.2(-6)

TABLE 11.14

AVERAGE Δp VALUES OF ION GAGES

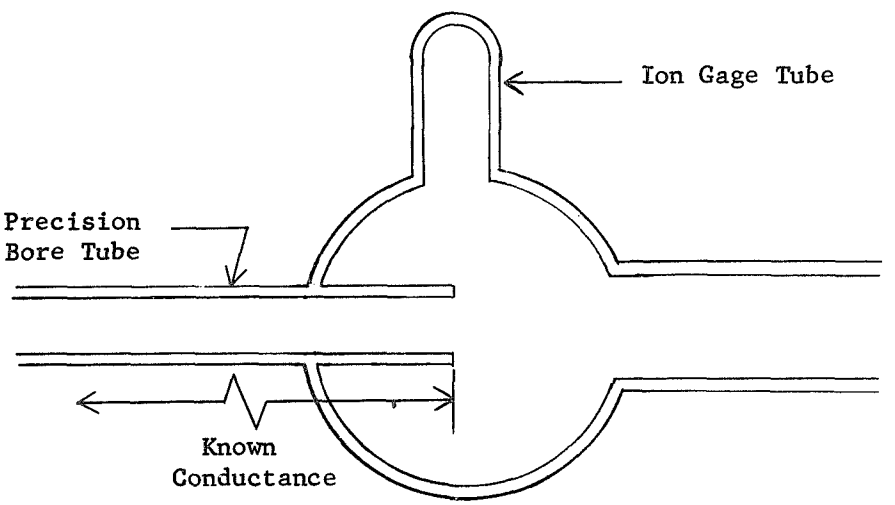
	G A S E S		
Gas	P	S	Q
CO_2	11.17	15.7	15.73
O_2	5.87	8.43	8.1
H_2	4.8	6.83	6.5
CO	9.0	12.9	12.93
N_2	8.63	12.2	12.23
H_2O	4.67	7.3	7.0

NOTE: Multiply above by 10^{-6}

11.3 Calculation of System Conductance

Section 11.3 is taken, in part, directly from "Contamination Exposures of Columbium and Tantalum Alloy Specimens", final report, period February 25, 1965 to June 15, 1966, by R. B. Fleming. This was done to add completeness to this report since the analysis is applicable to the program covered in this report.

The conductance between the two flow measuring gages consisted of a specially prepared length of precision bore tubing. In order to avoid possible uncertainties in conductance at the ends, the ends were prepared as shown below.



Thus, the precision bore was carried all the way to the end of the conductance, where there was an abrupt end with sharp edges. The conductance of this cylindrically shaped passage can be easily calculated by standard formulas which have been proven experimentally.

In addition, it was desired to avoid the possibility of any deviation from the cosine distribution for molecules leaving the walls, as the standard formulas are based upon this distribution. It has been reported (3) that some small deviations from cosine have been observed with certain molecules on glass. The inside wall of the conductance was, therefore, roughened by grit blasting to produce a frosty appearance. The grit blasting removes only a small amount of glass (about 0.01 mm) and it is believed that the roughened surface will assure a cosine distribution. The calculation of conductance is as follows. The most accurate equation takes into account the "clausing effect" (4) and can be written:

$$C_o = B(T_o/M)^{1/2}$$

where

$$B = 3.638\ K \frac{\pi D_o^2}{4}$$

For definition of symbols, see Section 3, "Nomenclature".

11.4 Analysis of Pressure Distribution

The installation of the third ion gage (identified as the "quartz" gage) measuring P_3 in the system, provided an additional means for estimating the pressure directly over the specimen. In earlier experiments, the specimen pressure (P_2) was estimated by knowing the pressure of the "sample" gage, P_1, calculating the pressure drop through the tube between the sample gage and the specimen and applying a thermal transpiration correction. In predicting specimen pressure (P_2) from P_3, it should be necessary to use only a thermal transpiration correction as there should be no net flow of gas between the specimen and the P_3 gage.

Unfortunately, there has generally been a very large discrepancy between the specimen pressure as predicted by the sample gage (P_1) and the quartz gage (P_3) calculations. For this reason a detailed analysis of the flows and pressures were undertaken. Such an analysis was presented in the last Final Report (period of 22 January 1964 to 22 January 1965, NASA Contract NAS 3-4169). An improved analysis will be presented herein.

First, the flow rate, Q, is defined by $Q = p\dot{V}$. Since the perfect gas law gives

$$p\dot{V} = \frac{\dot{m}RT}{M}$$

the flow rate can be expressed as

$$Q = \frac{\dot{m}RT}{M} \qquad (11.1)$$

Along a tube with <u>axial</u> variations in temperature, the <u>mass</u> flow rate ($\dot{m}$) remains a constant regardless of the temperature. However, the flow rate, Q, varies with temperature. If Q_o = the flow rate where the temperature is T_o, and Q_n = the flow rate where the temperature is T_n,

$$Q_n = \frac{\dot{m}RT_n}{M} = \frac{\dot{m}RT_o}{M} \quad X \quad \frac{T_n}{T_o}$$

or,

$$Q_n = Q_o \quad \frac{T_n}{T_o} \qquad (11.2)$$

An important assumption, which was justified in the Final Report referenced above, is that gas, as it flows in molecular flow through a tube, immediately comes to the temperature of the wall at any cross section. (This assumption is based upon the fact that most molecules bounce back and forth between tube walls, and only slowly travel down the tube in the direction of new flow.)

As a result of this assumption, a thermal transpiration relationship can be applied across the edge of the furnace, from pressures P_A to P_B (see sketch in Section 3., "Nomenclature"). This relationship can be written (4):

$$(P_A/P_B) = (T_1/T_2)^{1/2} \qquad (11.3)$$

The following two equations can be written from the definition of conductance, C:

$$Q_1 = C_1 \ (P_1 - P_A) \qquad (11.4)$$

$$Q_2 = C_2 \ (P_B - P_2) \qquad (11.5)$$

Equations 11.3, 11.4 and 11.5 can be combined, eliminating P_A and P_B to give:

$$P_2 = P_1 \left(\frac{T_2}{T_1}\right)^{1/2} - \frac{Q_1}{C_1}\left(\frac{T_2}{T_1}\right)^{1/2} - \frac{Q_2}{C_2} \qquad (11.6)$$

Equation 11.2 can now be used to put Q_1 and Q_2 in Equation 11.6 in terms of Q_o to yield

$$P_2 = P_1\left(\frac{T_2}{T_1}\right)^{1/2} - \frac{Q_o(T_1T_2)^{1/2}}{C_1T_o} - \frac{Q_oT_2}{C_2T_o} \qquad (11.7)$$

This equation gives the predicted pressure directly over the specimen as a function of the flow rate, temperatures, conductances and P_1. The conductance of a tube in molecular flow is given (4) by the following equations:

$$C_1 = 3.81 \frac{D^3}{L_1}\left(\frac{T_1}{M}\right)^{1/2} \qquad (11.8)$$

$$C_2 = 3.81 \frac{D^3}{L_2}\left(\frac{T_2}{M}\right)^{1/2} \qquad (11.9)$$

If equations 11.8 and 11.9 are substituted into equation 11.7, there results finally:

$$P_2 = P_1\left(\frac{T_2}{T_1}\right)^{1/2} - \frac{Q_o\, L\, (MT_2)^{1/2}}{3.81\, D^3\, T_o} \qquad (11.10)$$

where D is the diameter of the quartz tube containing the specimen, and:

$$Q_o = C_o\,(P_o - P_1) \qquad (11.11)$$

and where, for the 10^{-10} system

$$C_o = 0.172\,(T_o/M)^{1/2} \qquad (11.12)$$

Thus, the pressure over the specimen can be predicted from a knowledge of P_o, P_1, the various temperatures and the system geometry.*

On the other hand, the pressure P_2 can be obtained from P_3 by a simple thermal transpiration correction:

$$P_2 = P_3\left(\frac{T_2}{T_3}\right)^{1/2} \qquad (11.13)$$

11.5 Calibration of Secondary Standard Gages

In December 1965, the two secondary standard gages were sent to the AVCO Corporation, Wilmington, Massachusetts, for recalibration. Calibrations were obtained with both oxygen and nitrogen, in case nitrogen would be used later in the program.

The calibrations were obtained in the 10^{-7}, 10^{-6} and 10^{-5} torr ranges. The low 10^{-7} torr pressure was as low as the AVCO apparatus would permit. The estimated limit of error was ± 10%.

*The pressure over the specimen was assumed to be that existing at the center of the specimen. No attempt is made here to account for the presence of the specimen itself on the pressure distribution.

Results are presented in Figures 11.22, 11.23 and 11.24. One secondary standard gage tube is designated No. 10 and the other is No. 11. Each gage tube was always used only with its individual control panel. In order to reduce the possibility of inaccuracies, the secondary standard gage tubes and control panels were used only for calibration of the system gages and not for any other purpose.

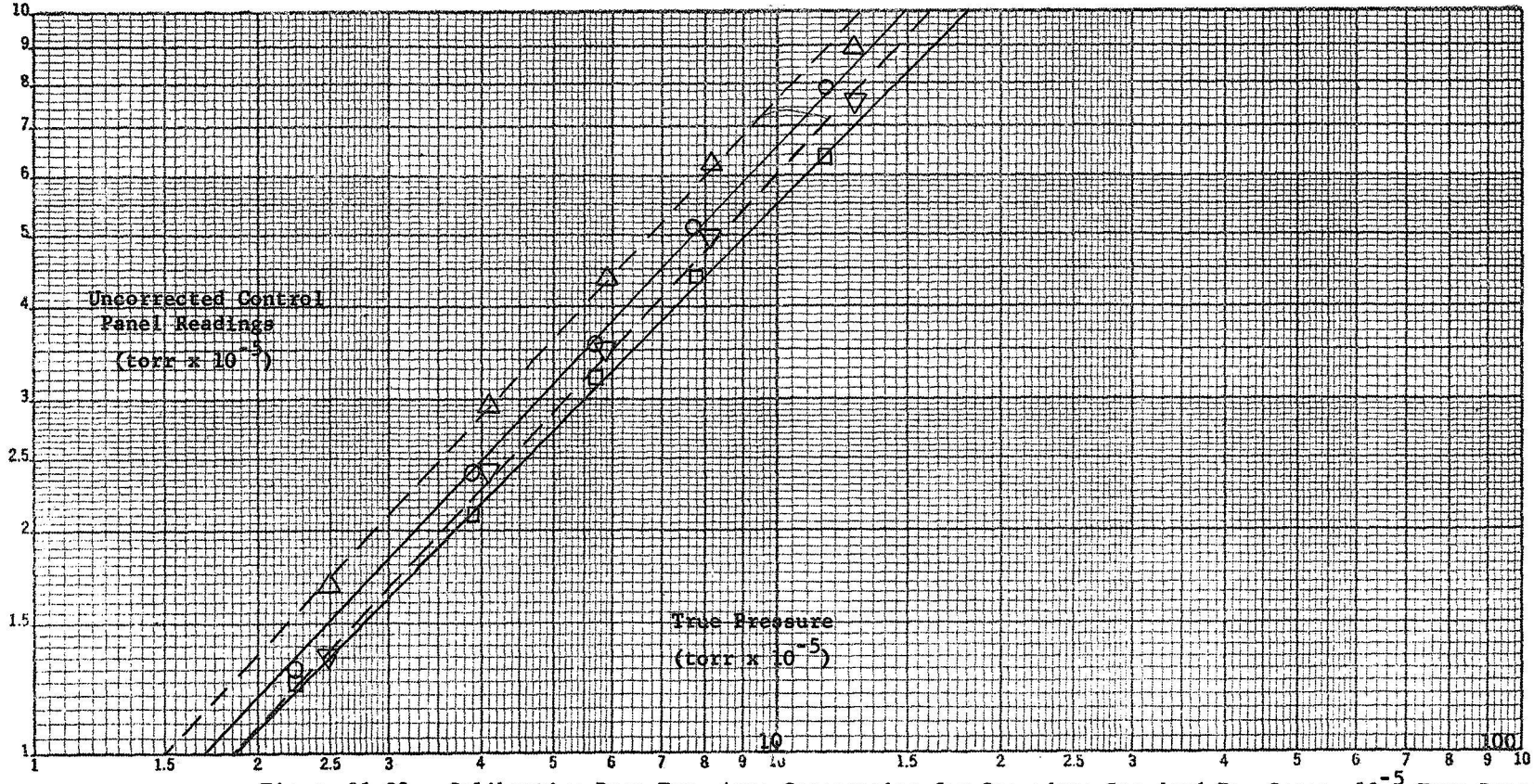


Figure 11.22 Calibration Data From Avco Corporation for Secondary Standard Ion Gages, 10^{-5} Torr Range

Dashed Lines: Ion Gage #10 With Control Panel #40002 ○ O_2 △ N_2

Solid Lines: Ion Gage #11 With Control Panel #30013 □ O_2 ▽ N_2

Emission Current: 1.0 mA

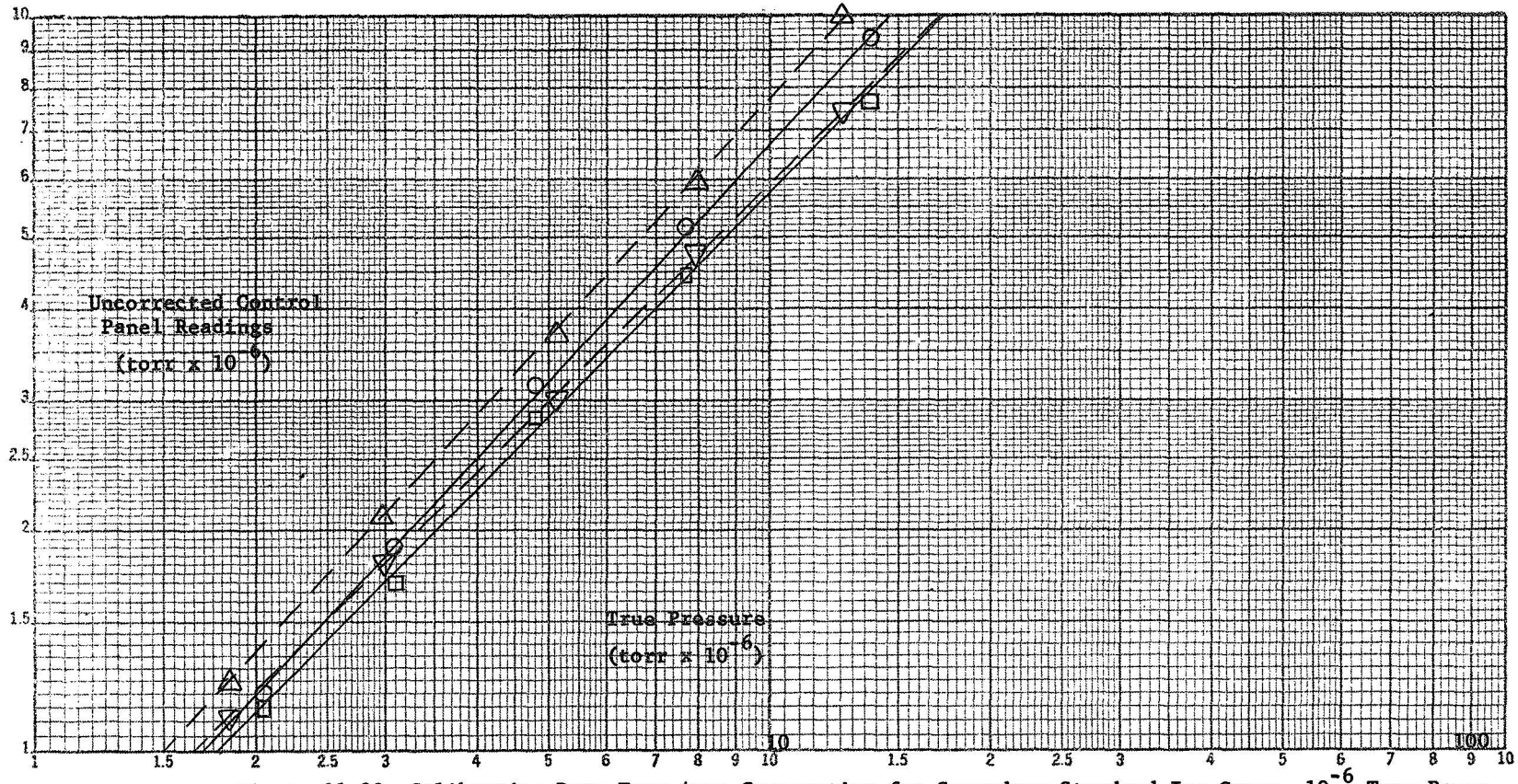


Figure 11.23 Calibration Data From Avco Corporation for Secondary Standard Ion Gages, 10^{-6} Torr Range.

Dashed Lines: Ion Gage #10 With Control Panel #40002 ○ O_2 △ N_2

Solid Lines: Ion Gage #11 With Control Panel #30013 □ O_2 ▽ N_2

Emission Current: 1.0 mA

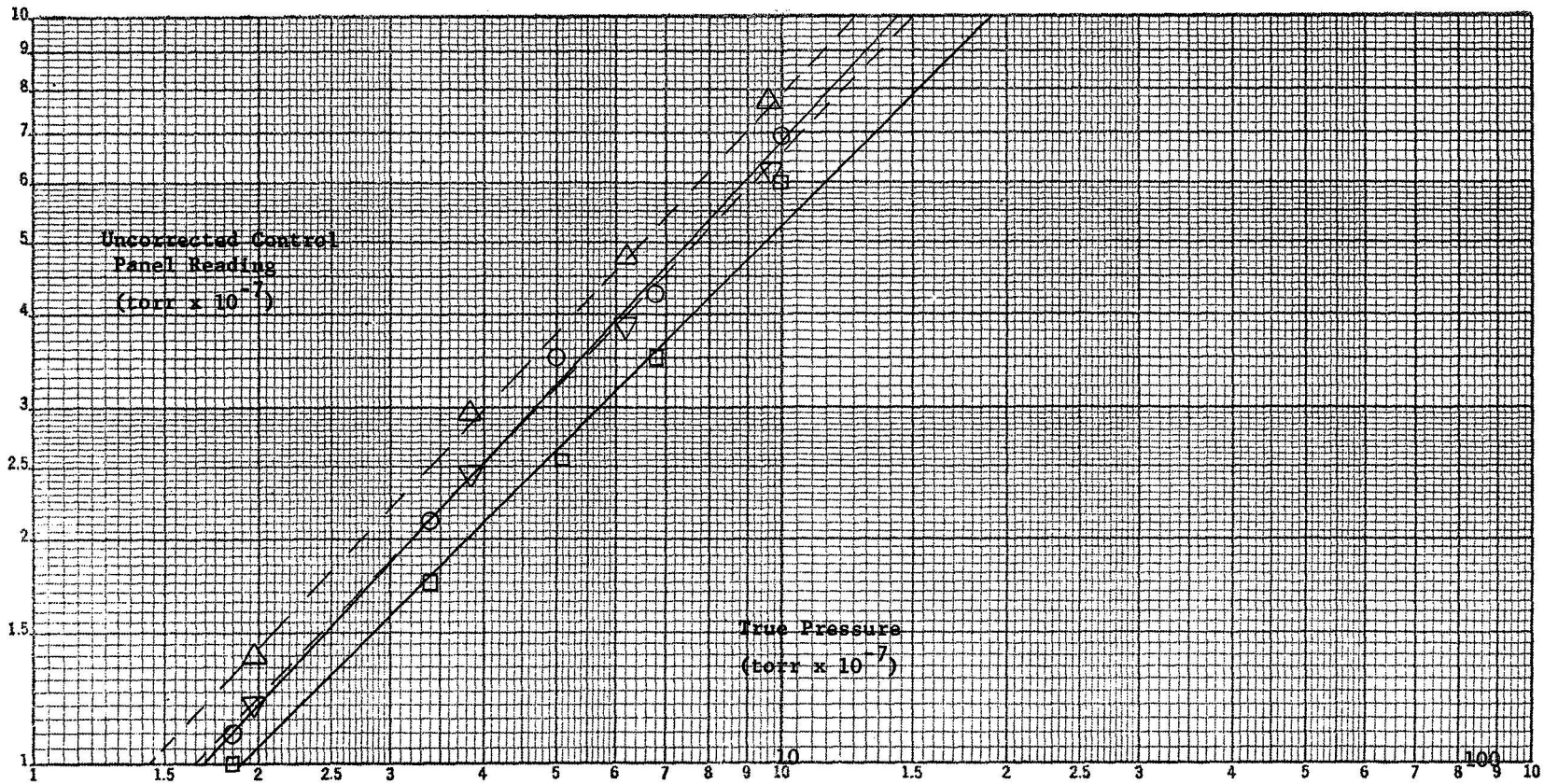


Figure 11.24 Calibration Data From Avco Corporation for Secondary Standard Ion Gages, 10^{-7} Torr Range

Dashed Lines: Ion Gage #10 With Control Panel #40002 ○ O_2 △ N_2

Solid Lines: Ion Gage #11 With Control Panel #30013 □ O_2 ▽ N_2

Emission Current: 1.0 mA

12.0 REFERENCES

1. BARRETT, C.A.: Absorption Rate Sticking Probabilities for Oxygen on Columbium and Dilute Columbium-Zirconium Alloys. NASA TN D 4885, November, 1968.

2. SANTELER, D.J., et.al: Vacuum Technology and Space Simulation. NASA SP-105, 1966.

3. MOORE, B.C.: Measurement of Vacuum in Nonuniform Temperature Environments. J. Vac. Sec. and Tech., Vol. 1, Sept.-Oct., 1964, pp.10-16.

4. DUSHMAN, S. and LAFFERTY, J.M.: Scientific Foundations of Vacuum Technique. Second ed., John Wiley and Sons, Inc., 1962.

13.0 LIST OF PREVIOUS REPORTS

The previous reports on this subject which were prepared by the General Electric Company for the National Aeronautics and Space Administration are listed below.

1. Contamination Exposures of Cb-1Zr Specimens, Final Report for NASA Contract NAS 3-1903, Covering the Period September 8, 1961 to June 22, 1962.

2. Contamination Exposures of Cb-1Zr Specimens, Final Report for NASA Contract NAS 3-1903, Covering the Period January 2, 1963 to July 3, 1963, Rept. No. GE63FPD306.

3. Contamination Exposures of Columbium and Cb-1Zr Alloy Specimens, Final Report for NASA Contract NAS 3-4169 Covering the Period January 22, 1964 to January 22, 1965, Rept. No. DMS65-2.

4. Contamination Exposures of Columbium and Cb-1Zr Alloy Specimens, Final Report for NASA Contract 3-4169 Covering the Period February 25, 1965 to June 15, 1966.

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